Fifth Quarterly Report

STUDY OF PROCESS VARIABLES ASSOCIATED WITH MANUFACTURING HERMETICALLY-SEALED NICKEL-CADMIUM CELLS

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MARCH, 1971 THROUGH MAY, 1971

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I

ABSTRACT

The following report describes the effort and results of the fifth quarter period of a program to determine and study the critical process variables associated with the manufacture of aerospace, hermetically-sealed, nickel-cadmium cells. During the period, the impregnation/polarization process variable study was brought to a close with the completion of a series of related experiments. The results of the experiments are summarized as follows:

(1) A scanning electron photomicrographic examination of impregnated plaques revealed a significant difference in the physical configuration of the impregnated active materials between positive and negative plaques and with different process conditions.

(2) A mechanical strength test of impregnated plaques using high levels of free acid (HNO_3) in the impregnation solution indicated the nickel matrix of the plaque was attack by the acid and resulted in a subsequent loss in strength.

(3) The cathodic polarization of impregnated plaques (to precipitate the active hydroxide form of material) in a caustic solution of KOH resulted in severe blistering of the plaque surface. This phenomenon, however, did not occur under the same conditions when NaOH was used as the caustic media. An experiment was devised to measure the volume and rate of gas evolved in the two processes. The results permitted the postulation the mechanism involves a high gas generation reaction deep within the nickel matrix of the plaques during the initial phase of polarization unique to the KOH solution.

(4) A semi-quantitative spectrographic analysis of impregnation/polarization process solutions indicated impurity levels increase with repeated solution use (mainly Ni and Cd from the slight solubility of the nickel plaques and the active materials). Within the limited number of experiments performed, the increase did not appear to be a serious problem.

During the subject period, a general characterization of cell separator materials was initiated. The major conclusion resulting from the characterization of materials is as follows:

(1) Scanning electron photomicrographic examination of the materials revealed that a micropolypropylene material (Hercules "Microweb") exhibited distinct physical differences from other materials tested.

(2) Chemical analysis for the extractable organic content demonstrated that nylon separator materials possessed more extractables than the polypropylene separator materials.

(3) A chemical analysis for the inorganic contents of the separator materials showed Pellon 2505 (Pellon Corporation) material exhibited an unusually high Zn impurity level.

TABLE OF CONTENTS

TABLE OF CONTENTS (CONTINUED)

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

The objective of this program is to develop a process procedure and control for manufacturing nickel-cadmium aerospace cells with reliable five (5) year life capability. In order to achieve these objectives, each component part will be investigated separately and collectively to determine the critical variables and related interactions.

The total program consists of four (4) distinct, yet interrelated phases. The first phase consisted of a detailed analysis of our procedures in conjunction with a review of pertinent literature of nickel-cadmium batteries to assess critical variables of the various processes that affect cell performance. The second phase will involve the evaluation and testing (verification) of the variables and their interrelation as determined in Phase 1. This will include a design of experiments to experimentally identify critical variables and to establish tolerances required for uniform performance. Phase 3 includes the detailed preparation of a Quality and Reliability assurance Program, Acceptance and Manufacturing Flow Sheets and a complete hardware specification that can be practically implemented in a cost effective manner. This specification will be patterned after "S-716-P-23, Interim Model Specification for High Reliability Nickel-Cadmium Spacecraft Cells." The Fourth Phase of the program will be to implement the results of Phases 1 through 3 on a production basis. This effort will "prove out" the conclusions and will establish both validity of concept and applicability to production equipment and overall operational capability. During this phase, the deliverable items of separation, positive and negative plates will be prepared. Also, 20 nickel-cadmium cells of 20 ampere-hour size will be manufactured to the developed procedure. Inspection levels will be 100% minimum and complete traceability maintained.

A. Background

The first quarter of this program was devoted to investigating the dry-sintering process used in manufacturing porous nickel plaque. A factorial experiment was designed to examine the sintered plaque characteristics as a function of the process variables. The data gathered from this experiment were analyzed using a step-wise multiple regression technique designed for use with the IBM 1130 computer. At the completion of analysis, plaques with different characteristics were selected for use in the impregnation factorial experiment.

The second quarter's work encompassed additional variability analyses of the data from the unimpregnated plaque experiments. This effort included the prediction of the variability of the plaque manufacturing process within the normal tolerance range of the equipment and was instrumental in determining the critical variables and their effects upon the process for the purpose of instituting new control limits. This was followed by a short production run of three (3) different types of plaques (high strength-low porosity, average strength-average porosity and low strength-high porosity). These plaques are being used for the impregnation studies of the experimental program. Also during the second quarter, major effort was directed toward the setting up of a separate facility for the impregnation, polarization, formation, washing and other items related to the cell assembly for the contract. An experimental program was designed to study impregnation, polarization, formation and washing.

During the second reporting period, work began on the preparation of specifications for separator, ceramic-to-metal seal cover assembly and unimpregnated plaque manufacturing control.

The third quarterly period included an investigation (strength tests and scanning electron micrographic analysis) of the properties of the three groups of sintered plaques produced for use during the impregnation/polarization study. This effort revealed an anomalous group of low strength which was attributed to a high sintering temperature in conjunction with a strong sintering furnace reducing atmosphere. This investigation also revealed a plaque group exhibiting characteristic highly desirable in a nickel-cadmium cell plaque; this included high strength and a very uniform distribution of the porous void.

The experimental data resulting from previously performed impregnation/polarization design experiments was analyzed to determine the effects of certain process variables which were selected for investigation in the present process study. This information was used during the following period to reduce the number of process variables considered for study in the new impregnation/polarization design experiments.

Preliminary drafts of Quality Assurance Specifications for sintered nickel plaques, separator materials, and ceramic-to-metal seals were also completed during this period.

The fourth quarter period of the program was devoted to the implementation and evaluation of the impregnation/polarization factorial design experiments formulated for the study of the associated process variables. A linear multiple regression analysis of the experimental data and the subsequent interpretation demonstrated the critical variables associated with the process. In addition, the subsequent interpretation permitted an assessment of the effects of the process variables upon the final product. The value of the knowledge gained from this phase of the program was substantiated by incorporation into specifications controlling the present production process.

With the completion of the fourth quarterly period which was originally intended as the last period, it was readily evident that many of the original program objectives had not been obtained. In view of this difficulty, the program running time was extended resulting in subsequent quarterly periods.

The following report summarizes the efforts and results of the program during the fifth quarter period. The first section describes four tests which are a continuation of and the completion of the impregnation/polarization proven variable study. The tests include:

(1) A scanning electron photomicrographic analysis of impregnated plaques to determine physical characteristics.

(2) A mechanical strength test of impregnated plaques to determine the effects of the use of high free acid levels in impregnation.

(3) A study of the mechanism which results in the more destructive effect upon impregnated plaques with the use of KOH or a polarizing media.

(4) A semi-quantitative spectrographic analysis of impregnation/polarization process solution to evaluate the impurity level changes with repeated use of the solutions.

The final section of this report introduces a new phase in the process control study program. The initial work in a study of cell separator materials is undertaken with an attempt to characterize a selection of the available materials. The preliminary characterizations process includes a scanning electron photomicrographic analysis for physical attributes, and a series of chemical analyses for organic and inorganic contents.

II. CONTINUATION OF IMPREGNATION/POLARIZATION STUDY

A. Scanning Electron Micrographic Analysis

Selected positive and negative impregnated plaques resulting from the previous impregnation/polarization process study (see Section II of the Fourth Quarterly Report[1]) were subjected to a scanning electron micrographic analysis. The purpose of this analysis was an attempt to gain comparative knowledge concerning the active material distribution in the pores of the sintered nickel plaques, the remaining porosity of the impregnated plaques, and the general overall quality of the impregnation as associated with the various impregnation/polarization process conditions. All impregnated plaques included in this phase of the study were processed through completion of the formation operation and may be considered in a state ready for fabrication into cells.

A plaque was selected from each of the following positive impregnation/polarization experiments (individual experiments which constitutedthe design experiments described in the previous Quarterly Report) for analysis. The reasons for their selection are also stated below. A complete definition of the process conditions or levels associated with each experiment may be found in Appendix A of this report.

1. Experiment Number One - (Micrographs 1, 2 and 3) - This experiment was selected because the plaques produced exhibited the roughest surface of all the experiments.

Major Process Conditions:

(a) Impregnation Temperature - 150°F

(b) Impregnation Time - 1 Hour

(c) Free Acid Content (Nitrate Solution) - 1 gm/liter

(d) Impregnation Under Vacuum - Yes

(e) Polarization Time - 1 Hour

2. Experiment Number 5A - (Micrographs 4, 5, and 6) - This experiment produced the best appearing plaques (smooth, clean surfaces) of the experiment performed.

Major Process Conditions:

(a) Impregnation Temperature - 200°F

(b) Impregnation Time - 1 Hour

(c) Free Acid Content - 1 gm/liter

(d) Impregnation Under Vacuum - No

(e) Polarization Time - 1 Hour

3. Experiment Number 6A - (Micrographs 7, 8 and 9) - Plaques exhibiting the greatest active material electrical efficiency were produced by this experiment.

Major Process Conditions:

(a) Impregnation Temperature - 200°F

(b) Impregnation Time - 1 Hour

(c) Free Acid Content - 4 gms/liter

(d) Impregnation Under Vacuum - No

(e) Polarization Time - 1 Hour

4. Experiment Number 9B - (Micrographs 10, 11 and 12) - This experiment was selected because it was similar to the above three experiments with the exception of a much shorter polarization time.

   Major Process Conditions:

   (a) Impregnation Temperature - 200°F

   (b) Impregnation Time - 1 Hour

   (c) Free Acid Content - 1 gm/liter

   (d) Impregnation Under Vacuum - No

   (e) Polarization Time - 15 Minutes

A plaque was also selected from each of the following negative impregnation/polarization experiments for analysis. A complete definition of process conditions or levels associated with each experiment may be found in Appendix B of this report.

1. Experiment Number 2 - (Micrographs 13, 14 and 15) - Plaques produced by the negative experiments did not exhibit the physical appearance differences that are associated with the positive experiments; this experiment and the following were selected because of their similarity in process conditions with the exception of polarization time.

   Major Process Conditions:

   (a) Impregnation Temperature - 150°F

   (b) Impregnation Time - 1 Hour

   (c) Free Acid Content - 0.5 gm/liter

   (d) Impregnation Under Vacuum - No

   (e) Polarization Time - 15 Minutes

2. Experiment Number 3 - (Micrographs 16, 17 and 18) -

Major Process Conditions:

(a) Impregnation Temperature - 150°F

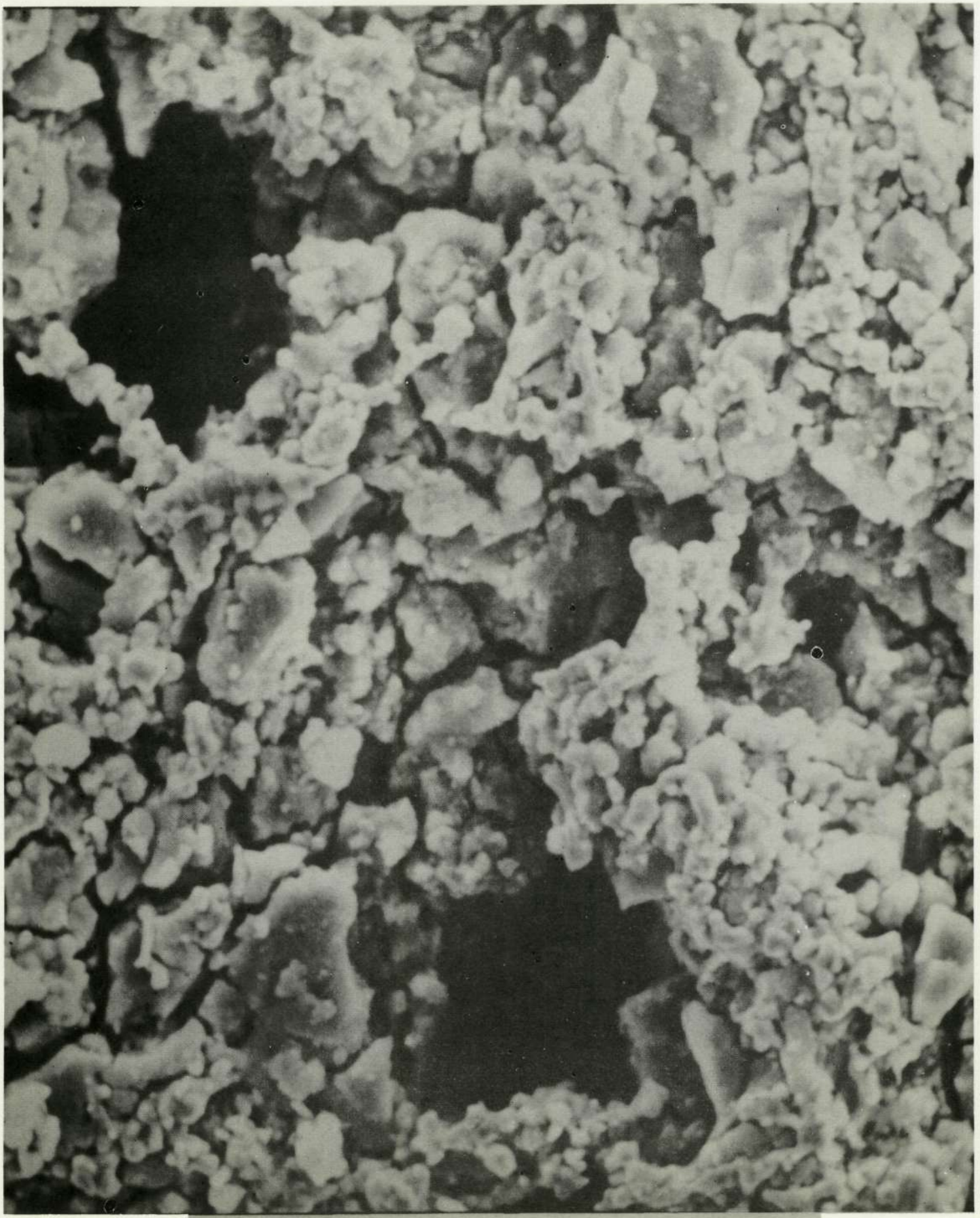
(b) Impregnation Time - 1 Hour

(c) Free Acid Content - 0.5 gm/liter

(d) Impregnation Under Vacuum - No

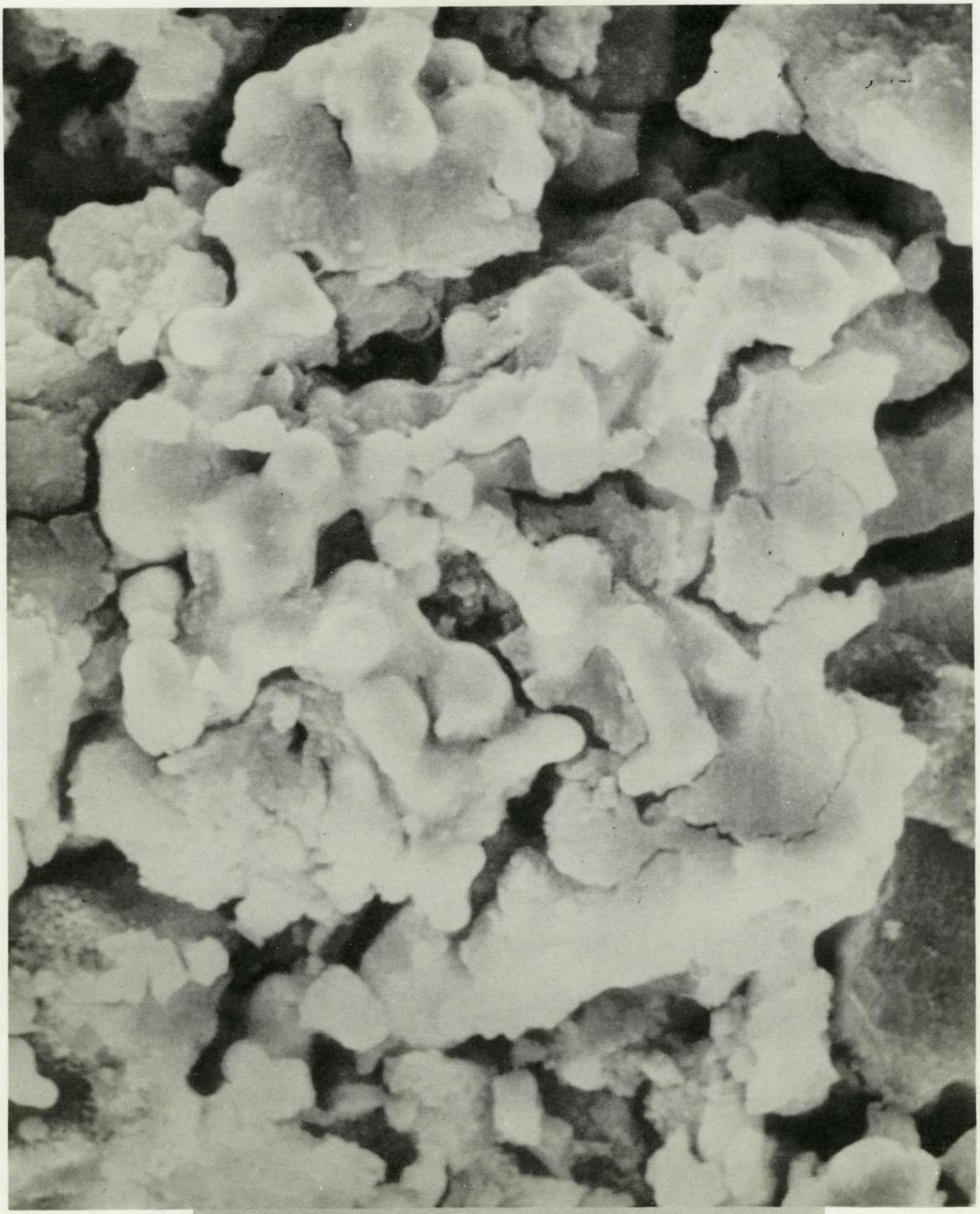
(e) Polarization Time - 1 Hour

The following photomicrographs are the result of the above analysis. Normally, three (3) shots are taken of each plaque sample of increasing magnification (1250X, 2500X and 5000X).



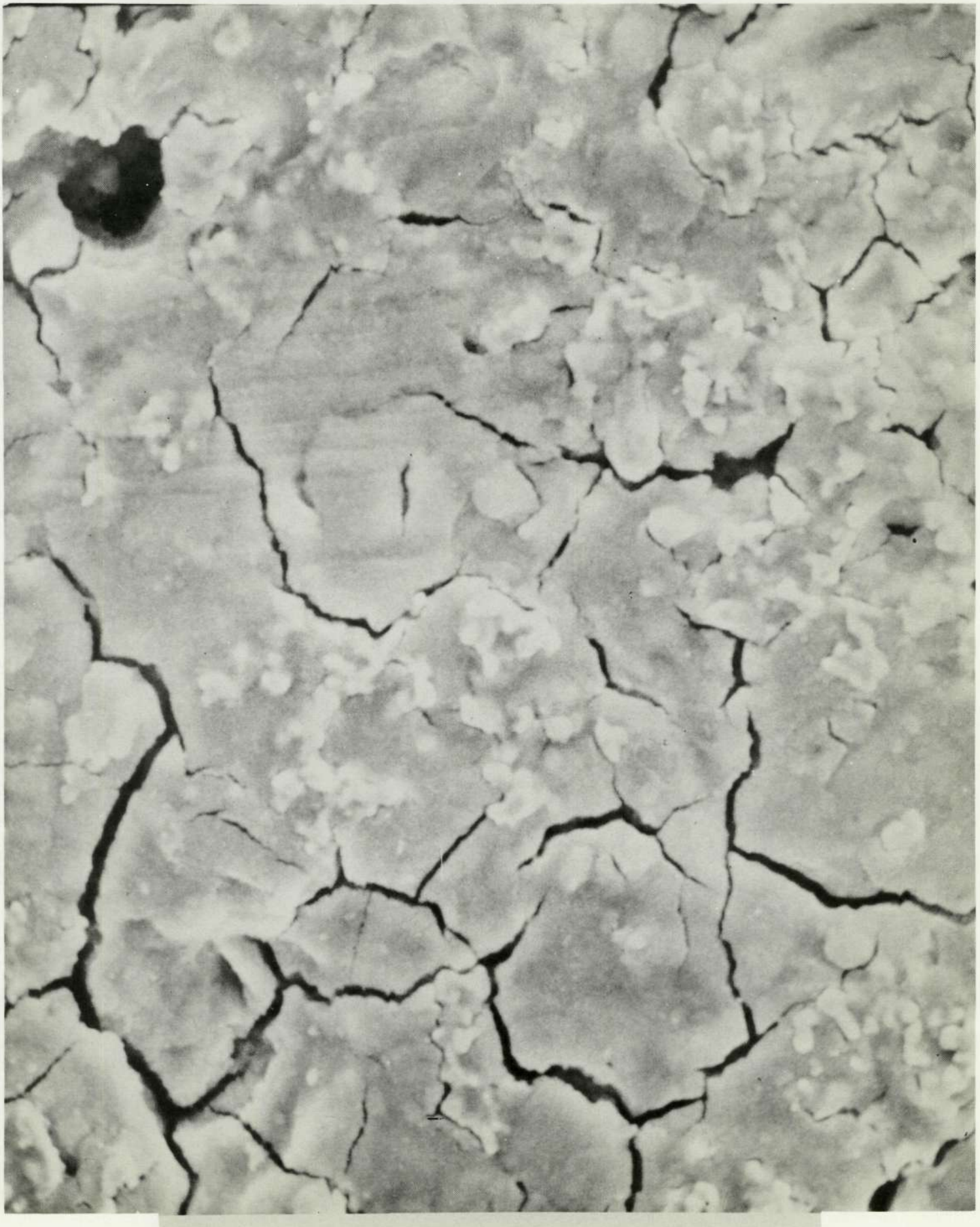
MICROGRAPH 1
Positive Plaque Experiment No. 1 - 1250 x Magnification

MICROGRAPH 2
Positive Plaque Experiment No. 1 - 2500 x Magnification



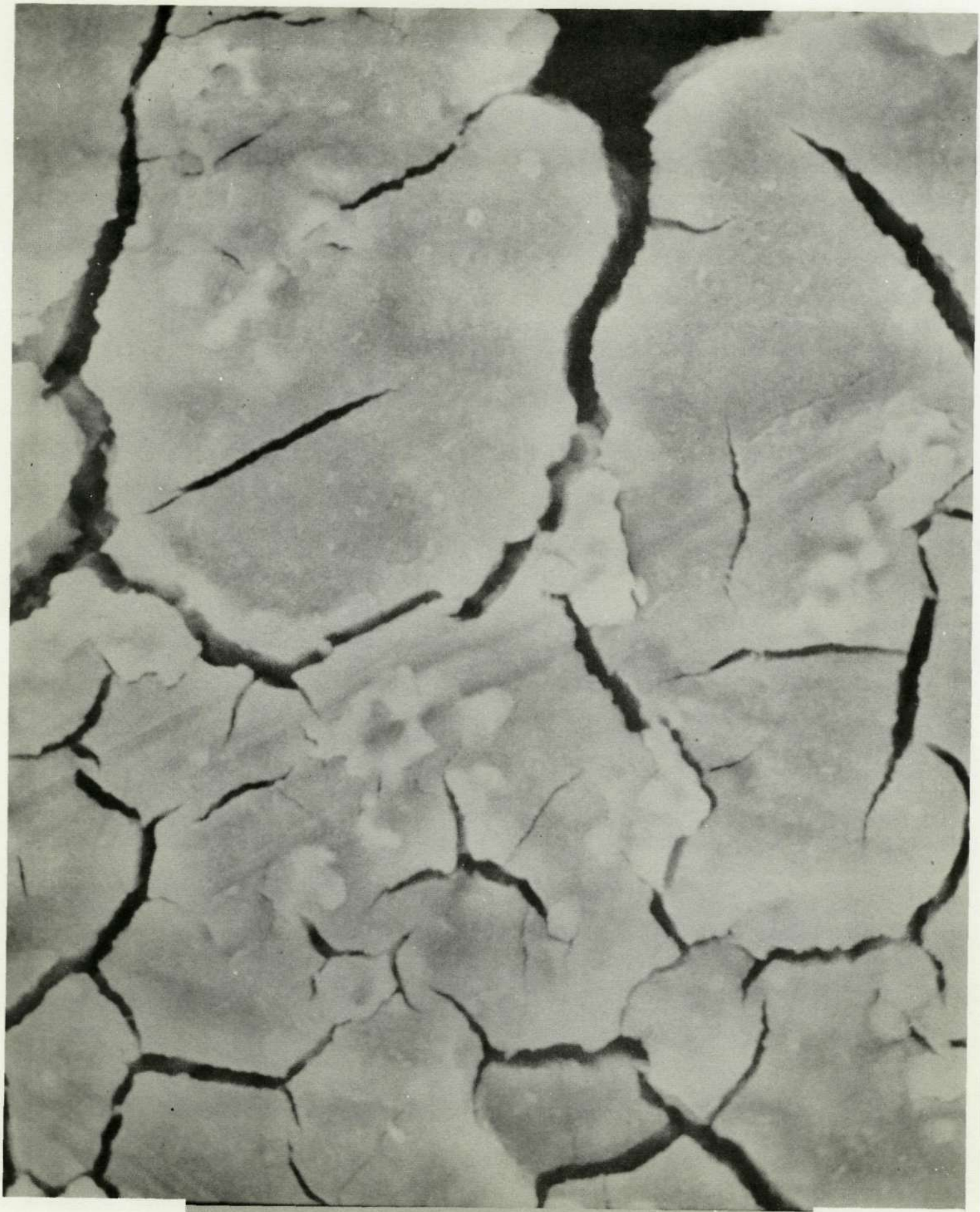
MICROGRAPH 3

Positive Plaque Experiment 1 - 5000 x Magnification

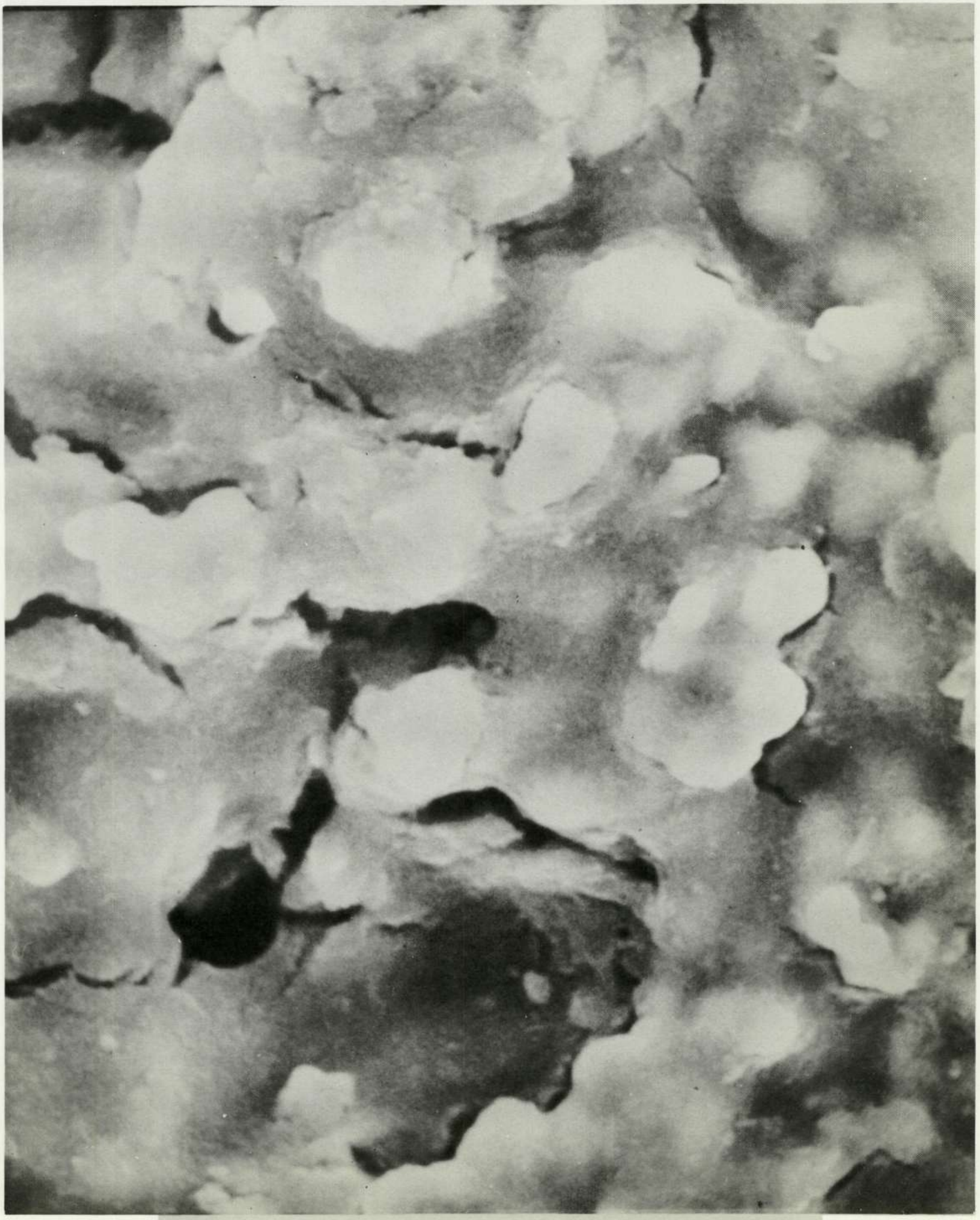


MICROGRAPH 4

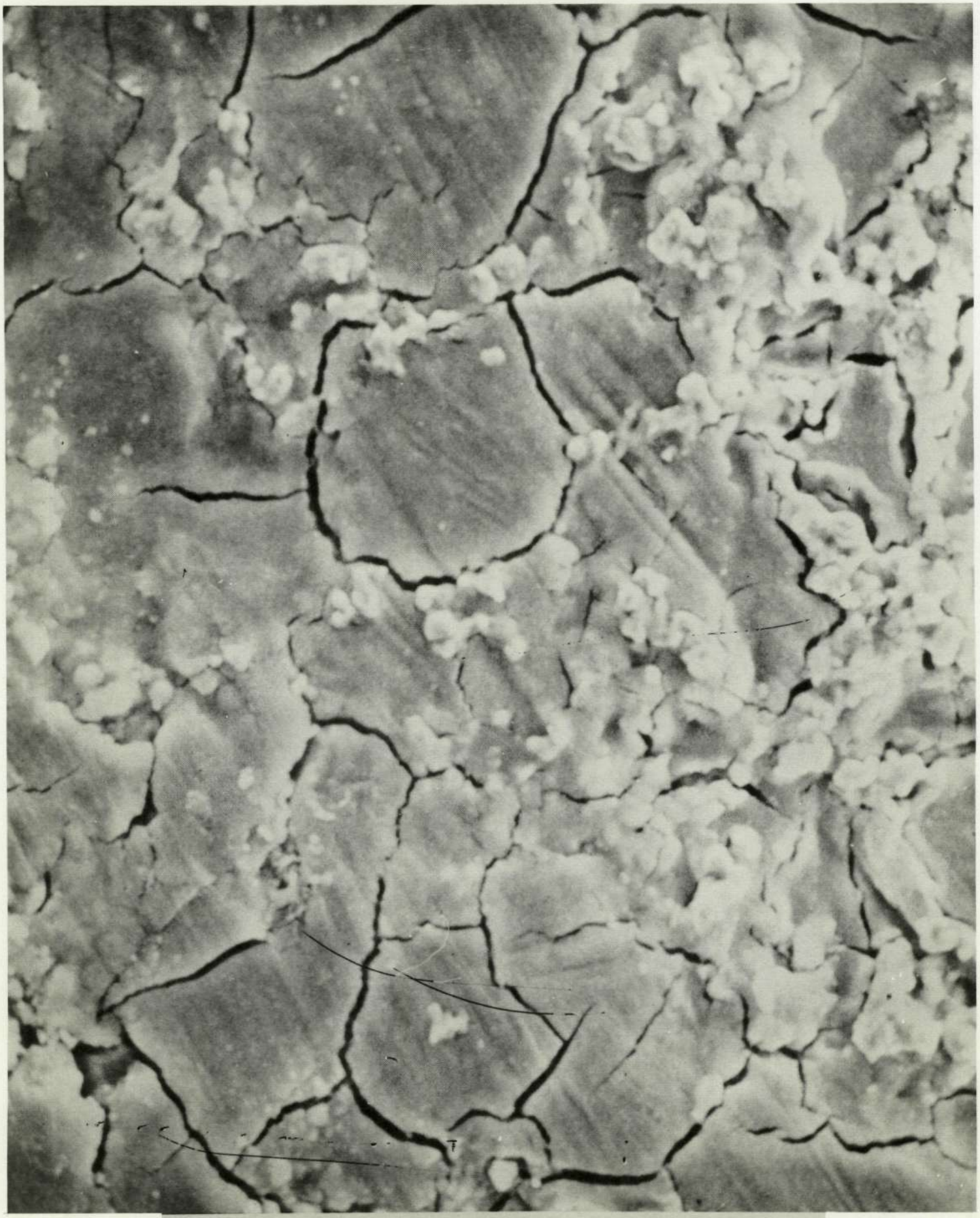
Positive Plaque Experiment No. 5A - 1250 x Maginification



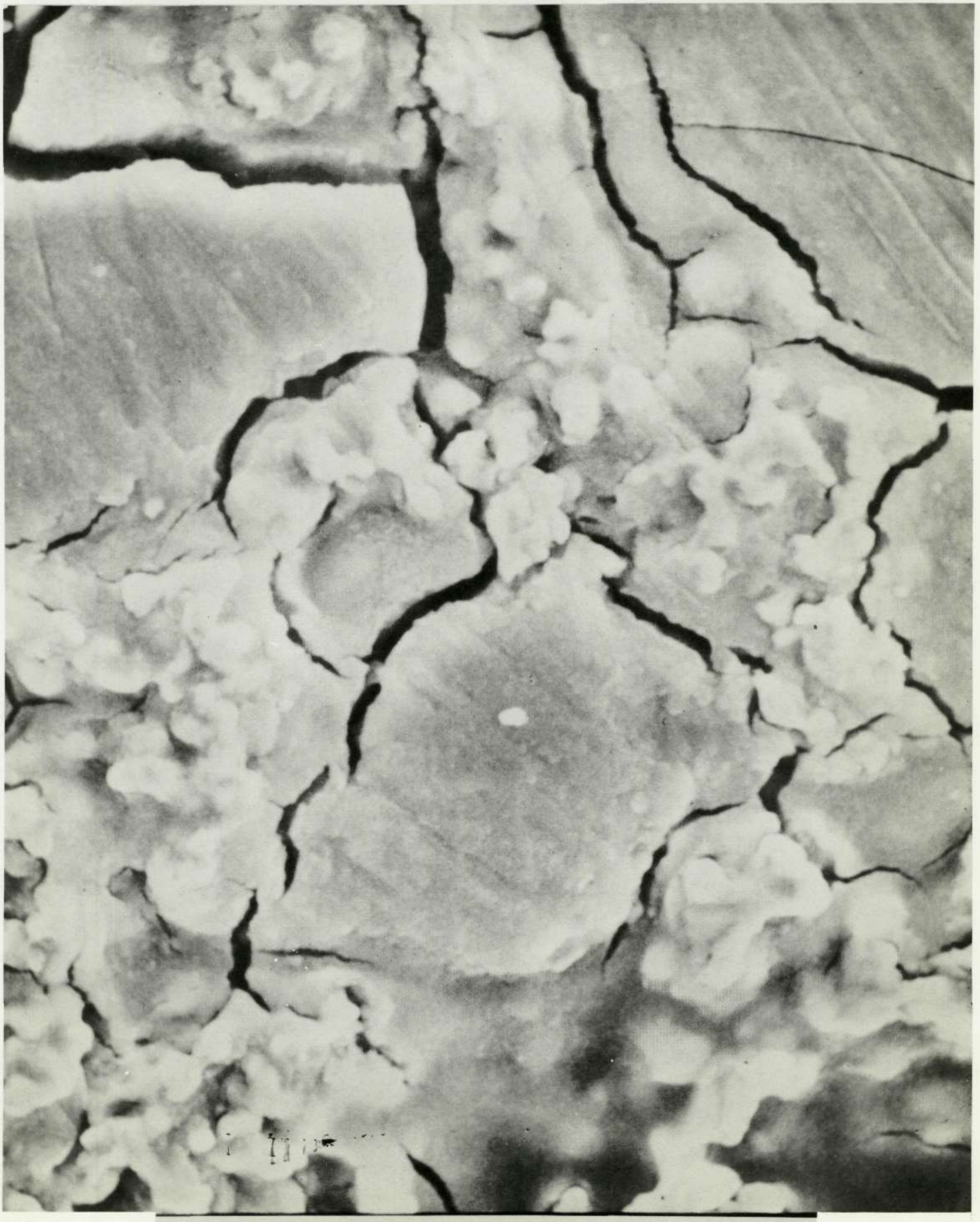
MICROGRAPH 5
Positive Plaque Experiment No. 5A - 2500 x Magnification



MICROGRAPH 6
Positive Plaque Experiment No. 5 A - 5000 x Magnification

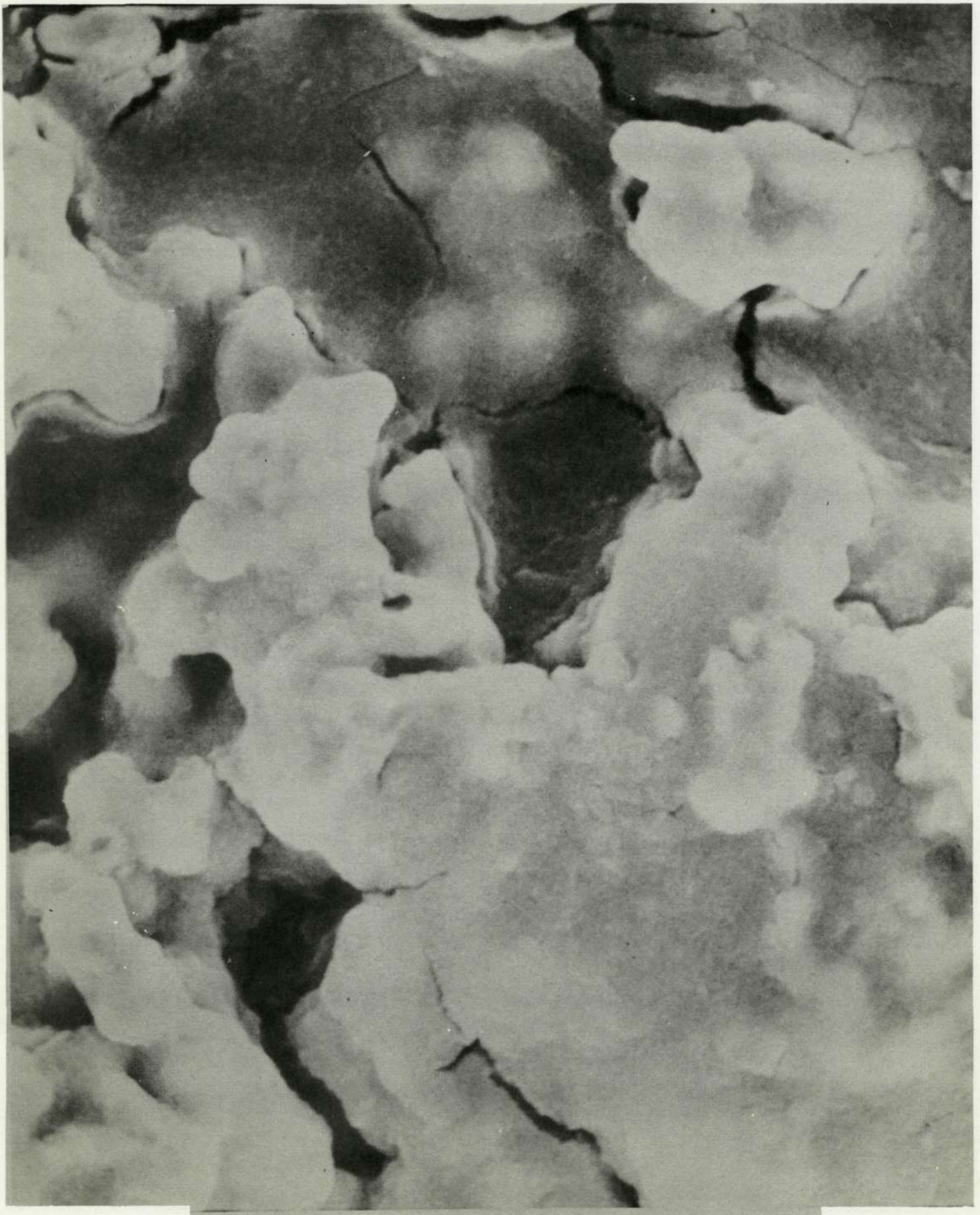


MICROGRAPH 7
Positive Plaque Experiment No. 6A - 1250 x Magnification



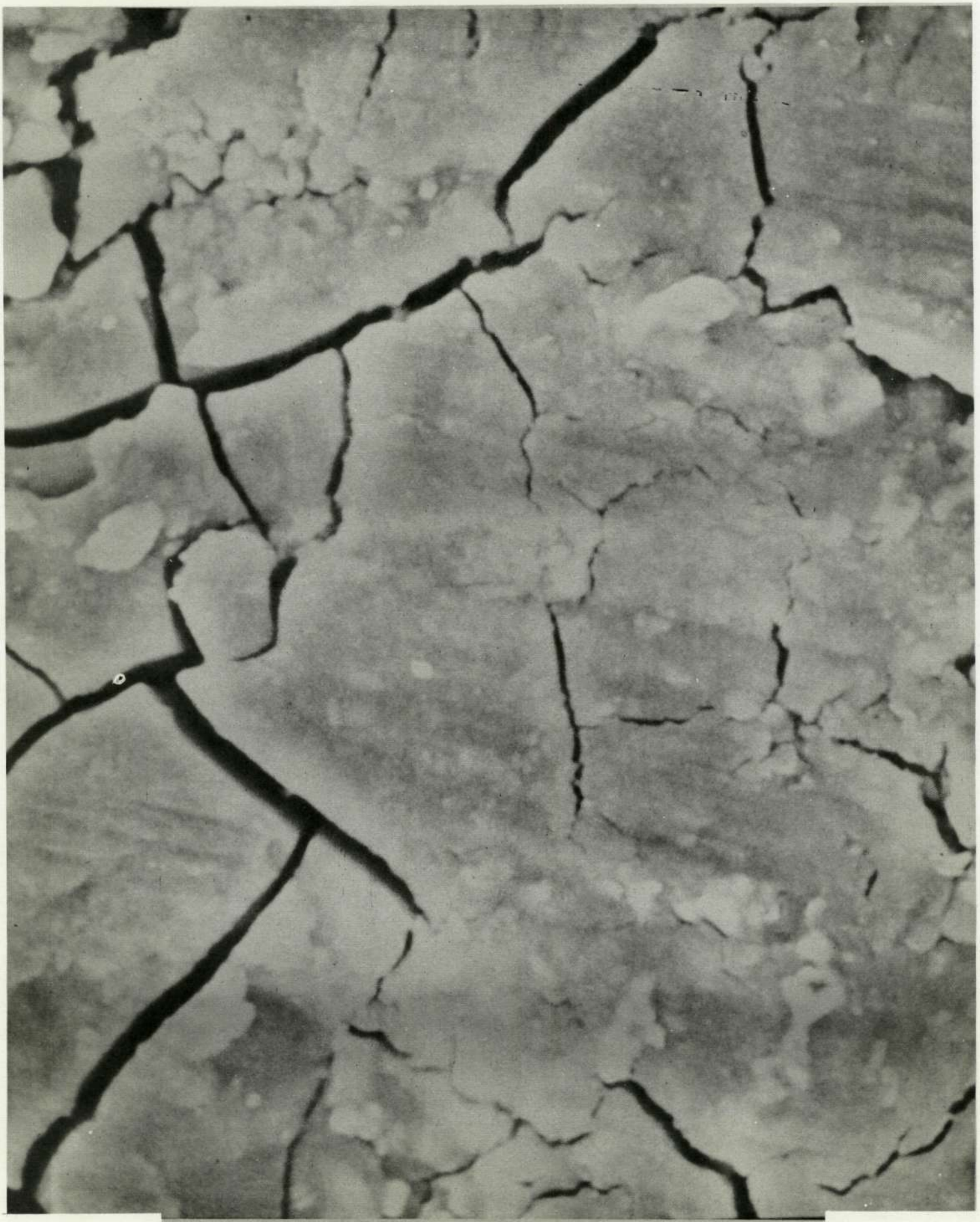
MICROGRAPH 8

Positive Plaque Experiment No. 6A - 2500 x Magnification



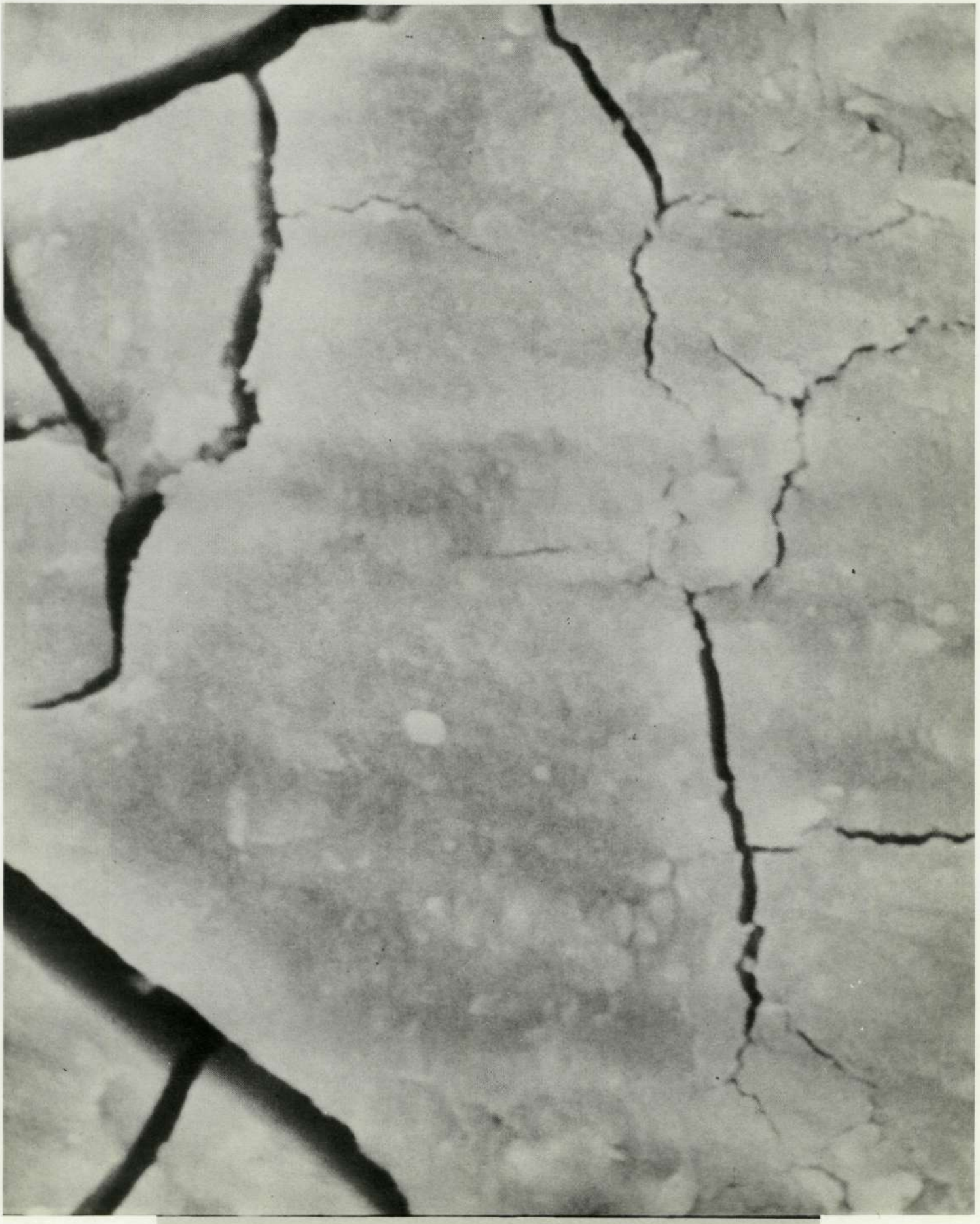
MICROGRAPH 9
Positive Plaque Experiment No. 6A - 5000 x Magnification

MICROGRAPH 10
Positive Plaque Experiment No. 9B - 1250 x Magnification

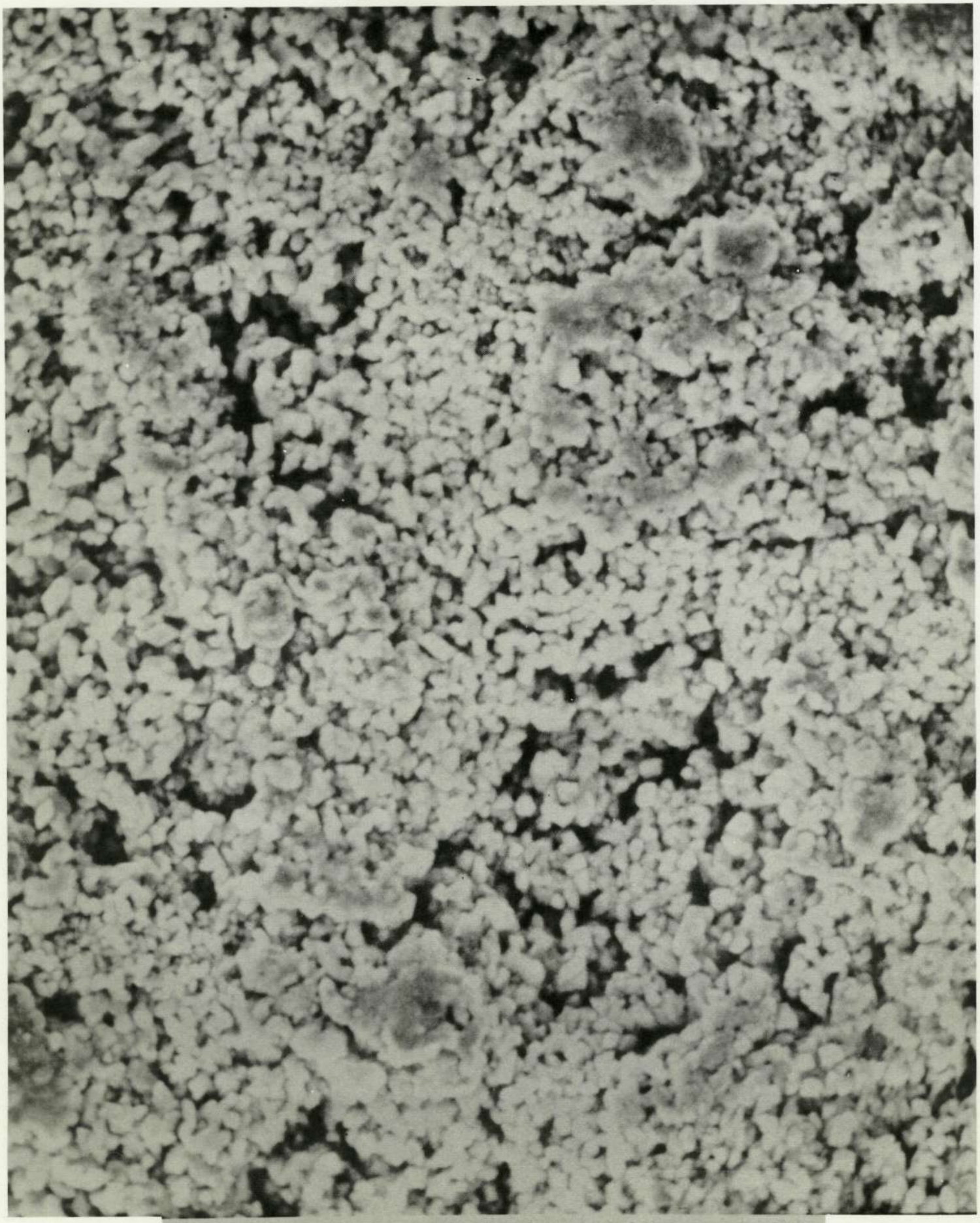


MICROGRAPH 11

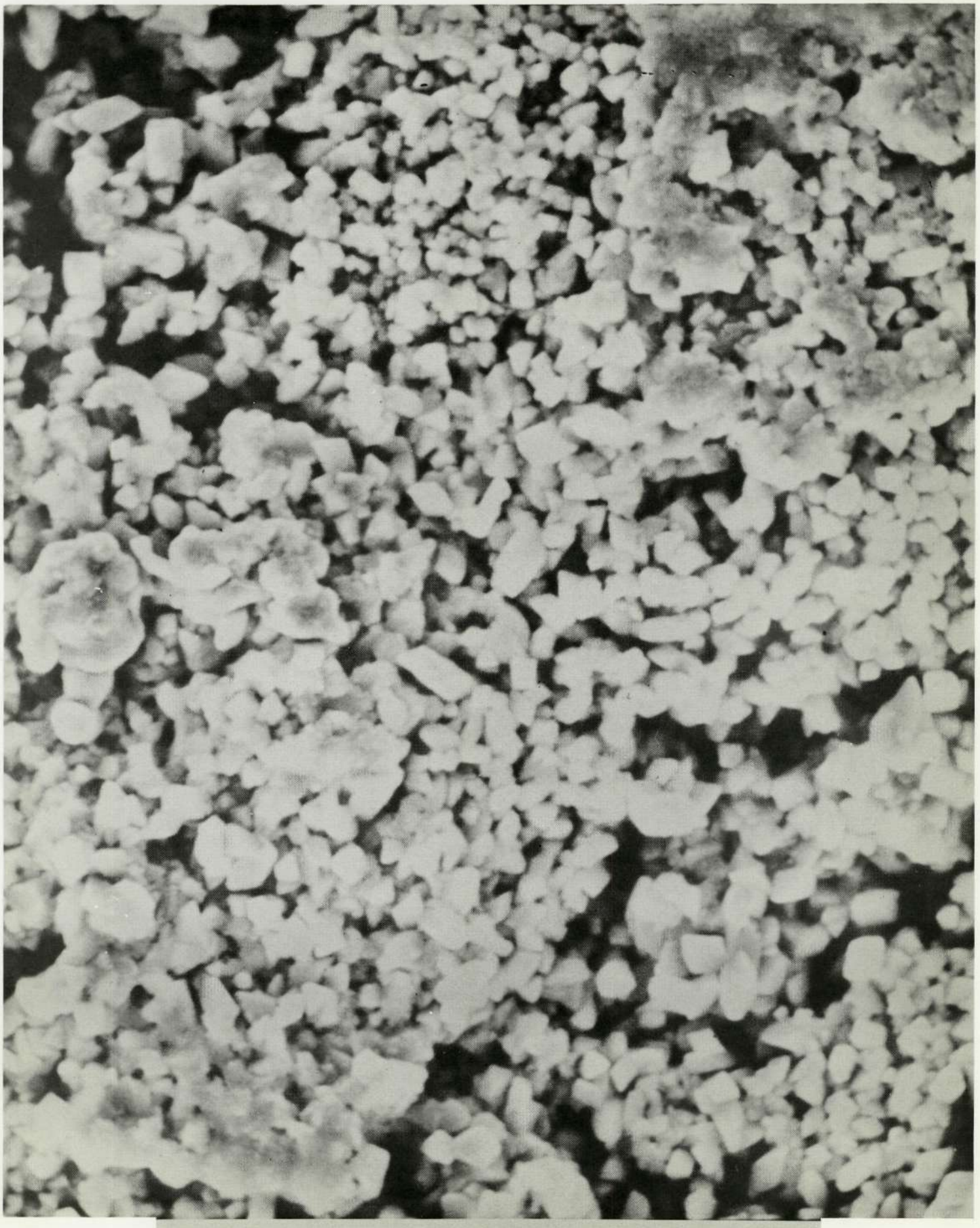
Positive Plaque Experiment No. 9B - 2500 x Magnification



MICROGRAPH 12
Positive Plaque Experiment No. 9B - 5000 x Magnification

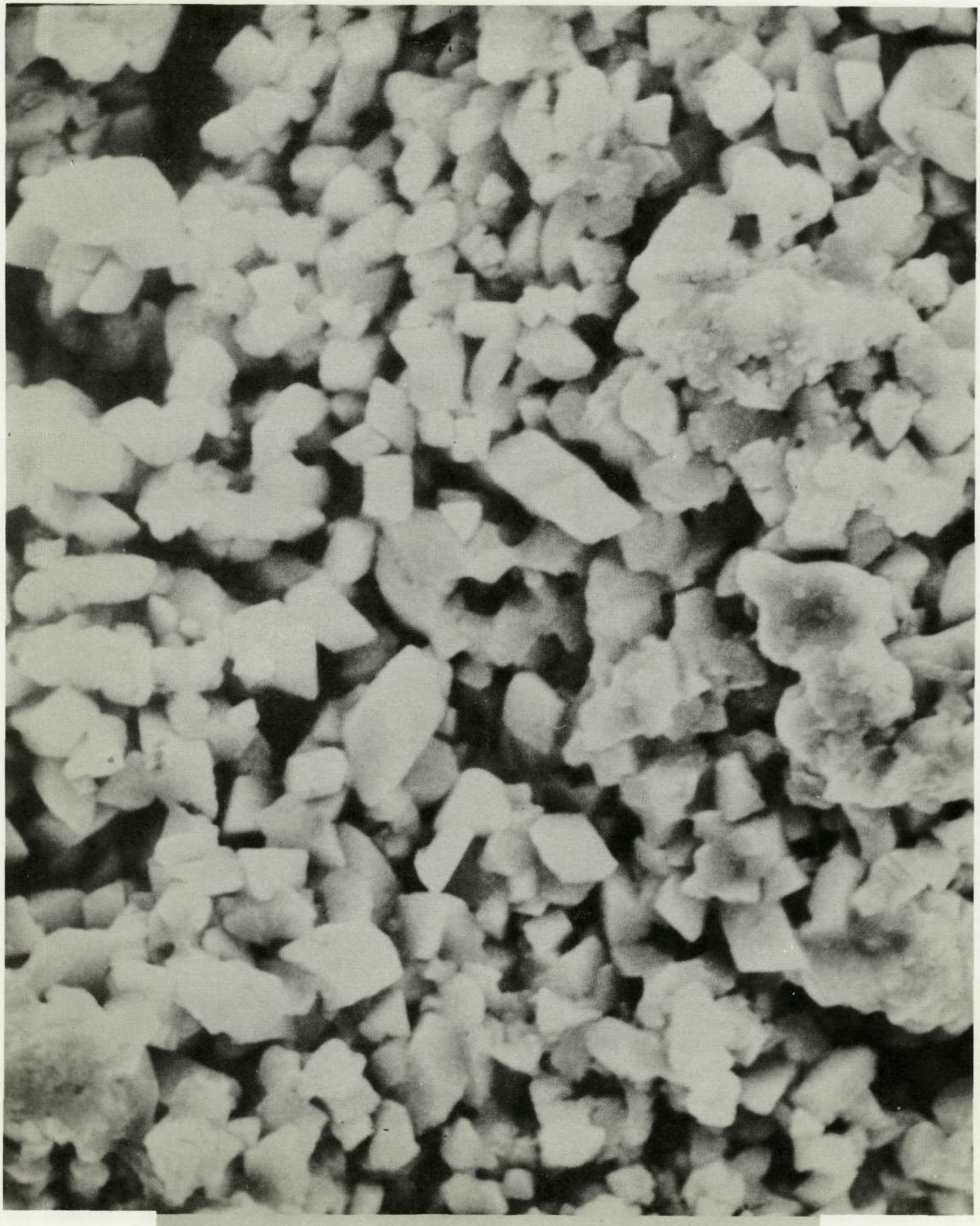


MICROGRAPH 13
Negative Plaque Experiment No. 2 - 1250 x Magnification

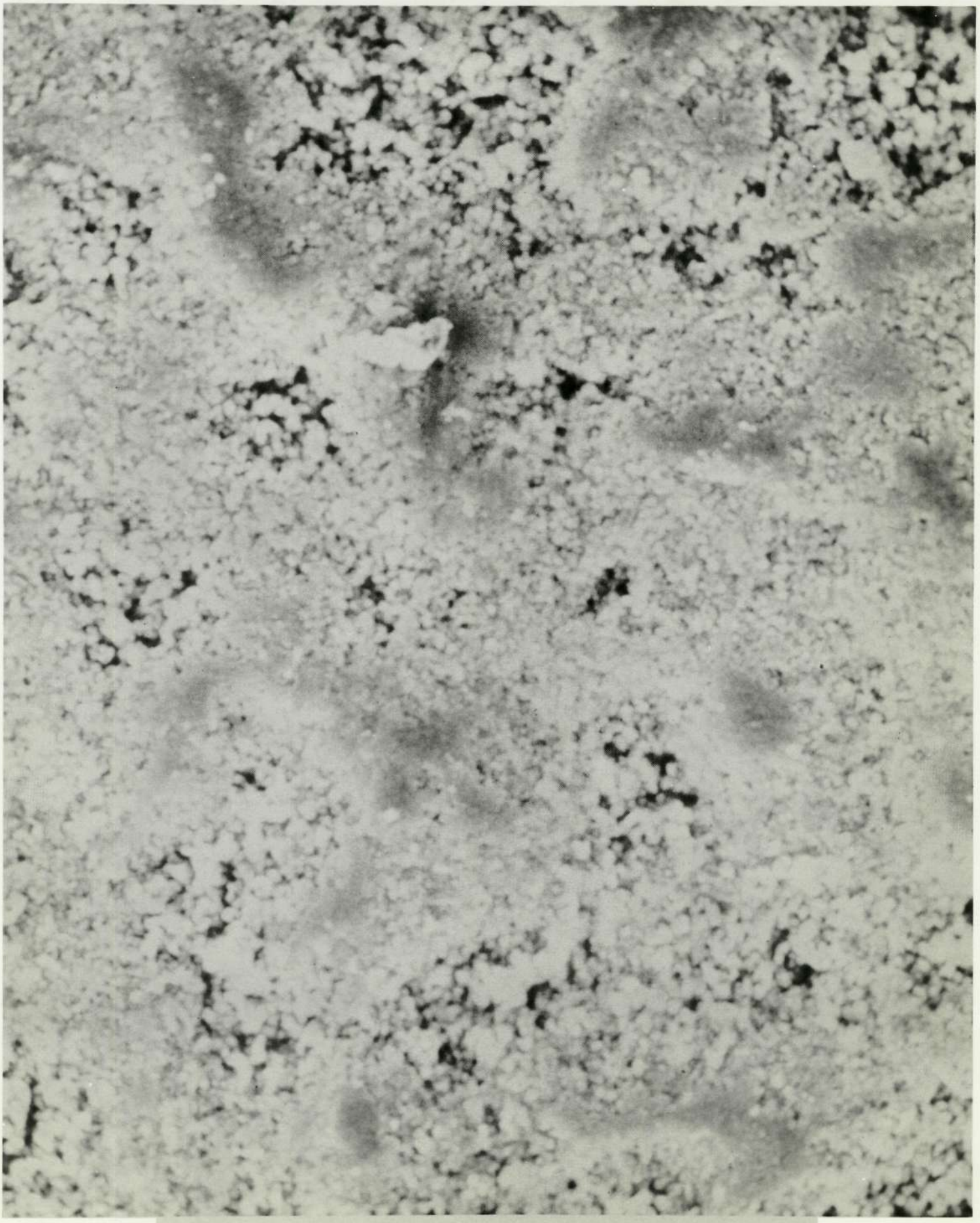


MICROGRAPH 14

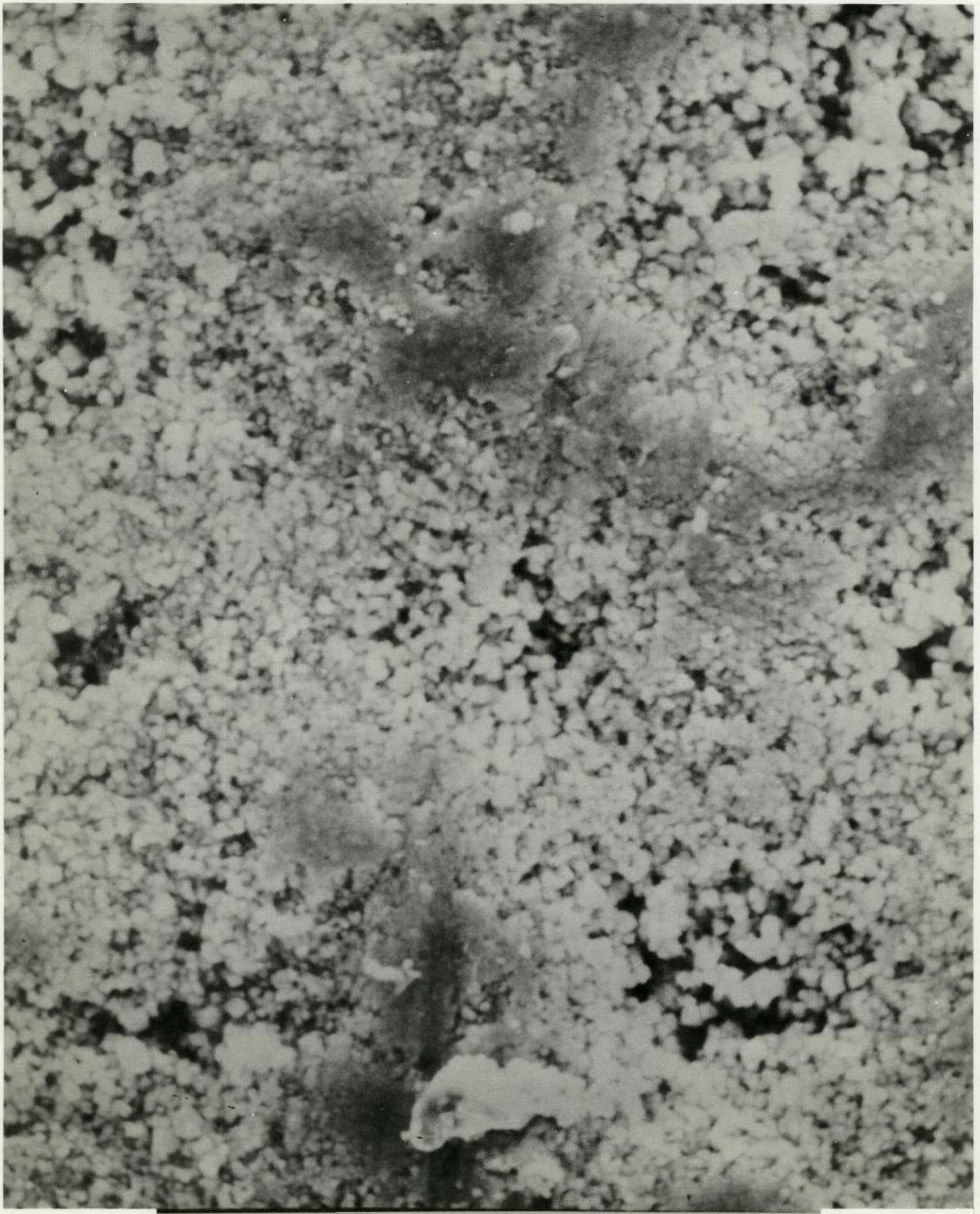
Negative Plaque Experiment No. 2 - 2500 x Magnification



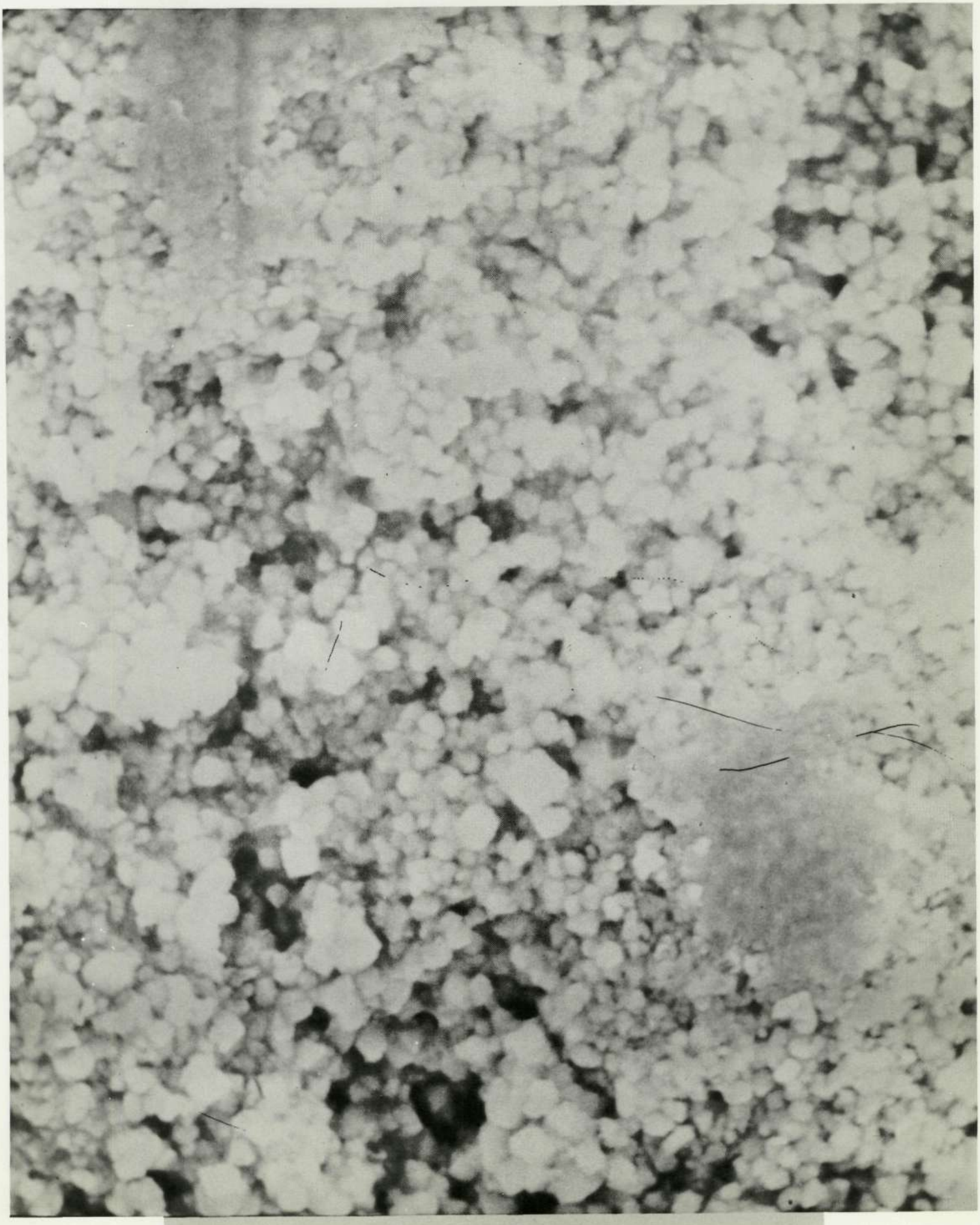
MICROGRAPH 15
Negative Plaque Experiment No. 2 - 5000 x Magnification



MICROGRAPH 16
Negative Plaque Experiment No. 3 - 1250 x Magnification



MICROGRAPH 17
Negative Plaque Experiment No. 3 - 2500 x Magnification



MICROGRAPH 18

Negative Plaque Experiment No. 3 - 5000 x Magnification

Observing the photomicrographs of the plaques from the positive experiments, the most distinguishing feature is the difference between Experiment Number One (Micrographs 1, 2 and 3) and the remaining positive experiments. The active material (gray area) in the plaque of Experiment Number One appears to be much more recessed and broken up in the nickel matrix (white area) and the impregnated plaque contains large voids (see Micrograph No. 1). In contrast, the remaining positive plaques, which exhibit very similar physical appearances, are much more "filled in" with little recession and no noticeable large voids. All plaques are impregnated with approximately the same amount of active material.

It might be assumed the more porous surface (easier accessibility of the electrolyte solution) of the Experiment Number One plaque would result in a superior electrical performance, but in actual electrical capacity testing of the subject positive plaques in a "flooded" electrolyte condition (see "Single Plate Capacity Testing", Section V, Third Quarterly Report[2]), the "filled in" or "caked" appearing impregnated positive plaques exhibited better capacities and active material efficiencies. These performances might be reversed in a "starved" electrolyte condition of a hermetically-sealed cell, but comparative data is not available.

Observing the photomicrographs of the plaque for Experiment Number 9B (Micrographs 10, 11 and 12), it is noticed there is little evidence of the protrusion of the nickel matrix as in the case of the similar plaques of Experiments 5A and 6A (Micrographs 4, 5 and 6 and Micrographs 7, 8 and 9) respectively.

This phenomenon is particularly noticed in the greater magnification, 5000X, of Micrographs 6, 9 and 12 in which the active material of Experiment Number 9B (Micrograph 12) appears to be less recessed into the nickel matrix. The basic process difference involved in Experiment Number 9B is its shorter polarization time (15 minutes). This would indicate that an extended polarization time (1 hour) results in a compaction effect or an increase in the apparent density of the active material.

In general, the positive impregnated plaque photomicrographs demonstrate the different impregnation characteristics which may be obtained by a change in the process conditions; a more porous, rougher plaque surface (Experiment Number One, Micrographs 1, 2 and 3) is associated with impregnation using a low nitrate solution temperature (150°F) under vacuum and the more "filled in", smoother appearing plaque surfaces are associated with impregnation using a high nitrate solution temperature (200°F) without vacuum (Experiment Number 5A, 6A and 9B, Micrographs 4 through 12).

Observing the photomicrographs of the plaques from the negative experiments, there is a readily observed difference in the make-up of impregnated active material in contrast to that which was observed in the positive plaques. The active material impregnation of the negative plaques is in the form of small crystal shaped particles with a few scattered patches of the "caked" appearing material associated with the positive plaques. Further observation will also disclose a difference in the particle size between the plaques of the two negative experiments selected for this analysis. Again, the plaques of both experiments exhibit approximately the same amount of active material impregnation.

The major difference between the experiments is the length of the polarization time. Negative Experiment Number 2 (Micrographs 13, 14 and 15) utilized a short polarization time of 15 minutes and as may be observed, the impregnated particles are relatively large and distinct. Negative Experiment Number 3 (Micrographs 16, 17 and 18), on the other hand, utilized a longer polarization time of one hour and as may be observed, the impregnated particles are much smaller. Polarization time appears to have an effect upon the size and shape of the active material particles in the negative plates.

Electrically, the finer particle plates exhibited greater capacities and efficiencies (see Table IV, p. 23, 4th Qtrly. Rpt.[1]), which could be attributed to the obvious greater surface area. With respect to recombination (oxygen reaction at the negative electrode-necessary in sealed cells), the plates exhibiting the larger particles might perform better (more porous surface to oxygen gas), but data is not available to support this postulation.

In general, the photomicrographs of the negative impregnated plaque indicates that longer polarization times reduce the size of the active material particles and improves the electrical capacity and efficiency of the negative plates.

B. Mechanical Strength Test

During the impregnation/polarization process study (previous Quarterly Report)[1], it was determined that superior electrical capacities and active material efficiencies were obtained in positive plaques impregnated in nitrate solutions containing high levels of free acid (4 grams/liter, HNO_3). This phenomenon was attributed to corrosion of the nickel matrix and the subsequent formation of additional active material. An obvious question then arose as to the effect of this treatment upon the mechanical strength of the finished plates. The following section describes an experiment which determined the relative mechanical strength of plates from two positive impregnation/polarization experiments which differed in levels of free acid used in impregnation.

The mechanical strength of the test samples was determined by a Four Point Bend Testing Machine described in detail in the First Quarterly Report[3] (see Section C). Basically, the procedure consists of punching a two (2) inch by one (1) inch sample from the test plate (the instrument for performing this task is also described in the above reference), supporting the test sample on two outer points and applying a compression load at two inner points (see Figure 1). A load cell senses the force applied to the supporting points and permits the determination of the force required for structural failure of the test sample.

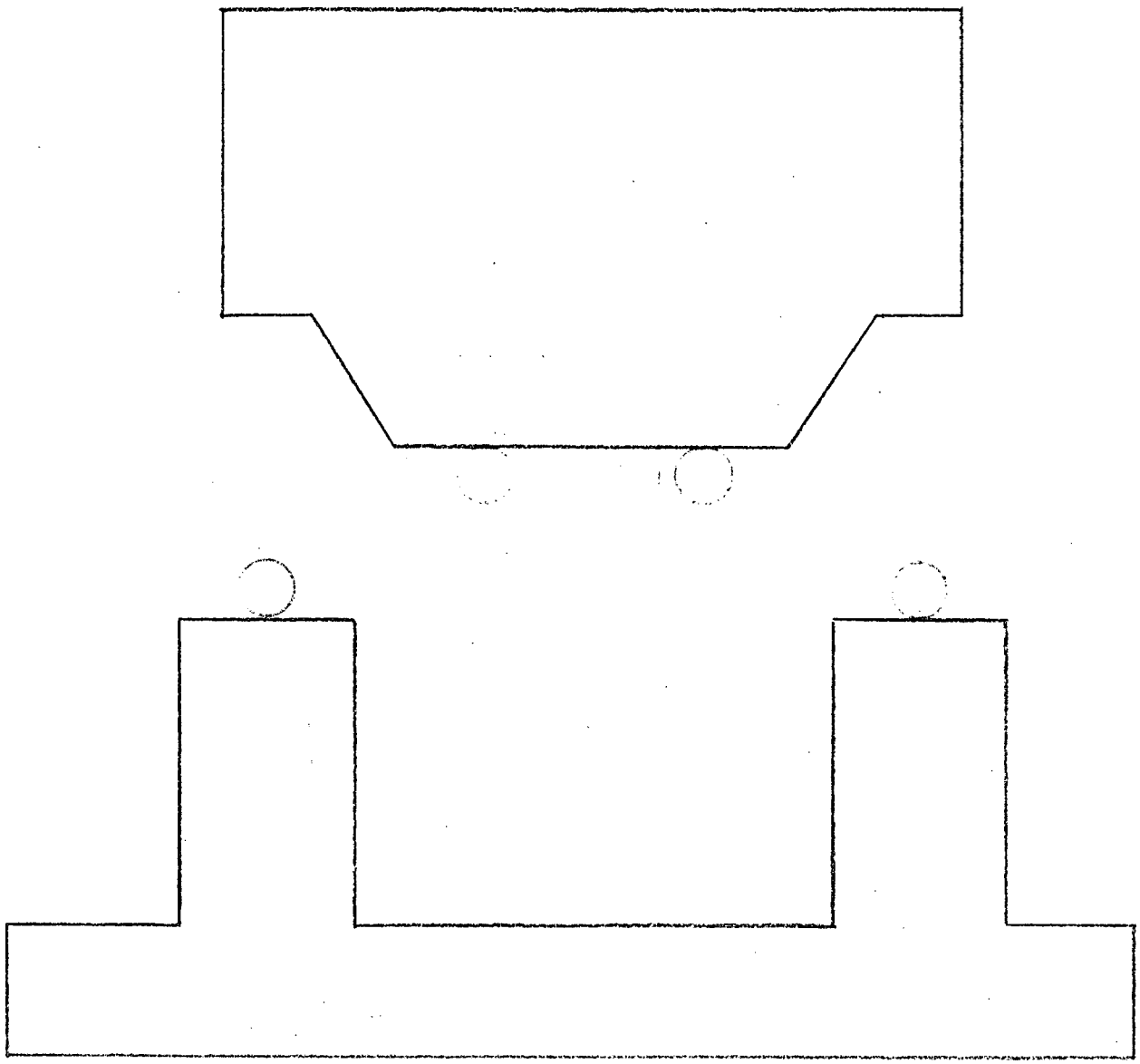


FIGURE 1

BENDING JIG

The experiments selected were positive Experiment 5A (nitrate solution free acid, 1 gram/liter) and positive Experiment 6A (nitrate solution free acid, 4 grams/liter). The plates produced in these two (2) experiments exhibited approximately the same amount of active material pick-up and were processed under similar variable levels with the exception of the free acid levels. As both of these experiments were also the subject of the preceding section, the specific experimental process data may be found therein.

Figures 2 and 3 present graphically the results of the mechanical strength tests. The graphs begin at a 0 load level; then as they enter two (2) points start to bend the test sample downward in a "U" shape, the pressure or load rises (downward movement of the recorder trace). Finally, at the point where the test sample starts to give with the applied pressure and the load tracing levels off, this is taken as the point of structural failure of the test sample. Each line of the recorder paper represents a force of 10 grams. The total force in grams required to cause structural failure is then determined by the distance of the recorder deflection. The stress in psi may also be calculated by use of the formula described on Page 13 of the First Quarterly Report.(3)

Two (2) tests were performed on a plate from each experiment. The first tests on Experiment 5A (see Fig. 2) have resulted in structural failure values of 340 grams (the 390 value indicated is in error) and 320 grams for an average value of 330 grams. The second tests on Experiment 6A (see Fig. 3) resulted in values of 300 grams and 280 grams for an average of 290 grams. In addition, it is observed there

Test #1

Test #2

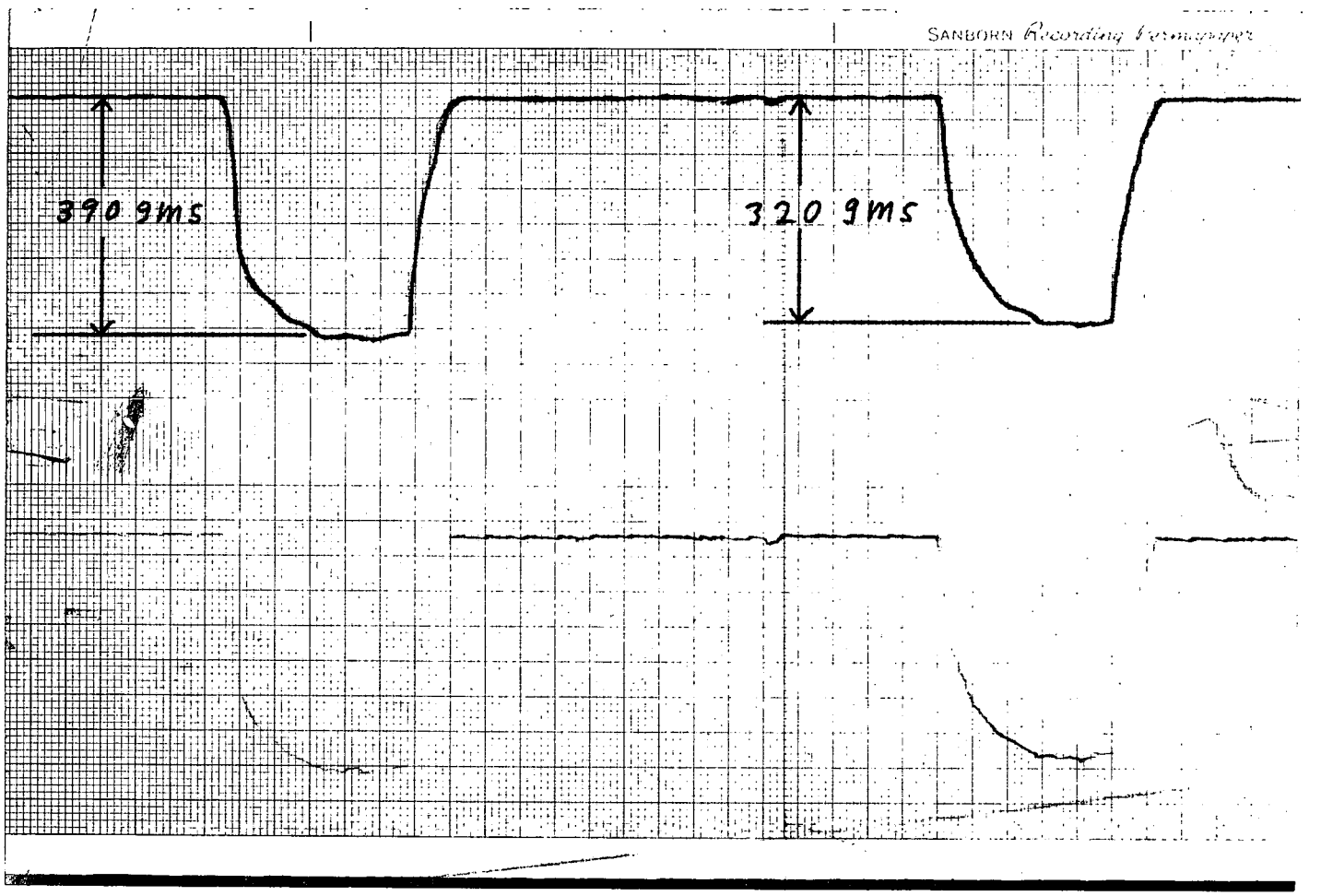


FIGURE 2

Mechanical Strength Test

Positive Plaque Experiment No. 5A

Test #3

Test #4

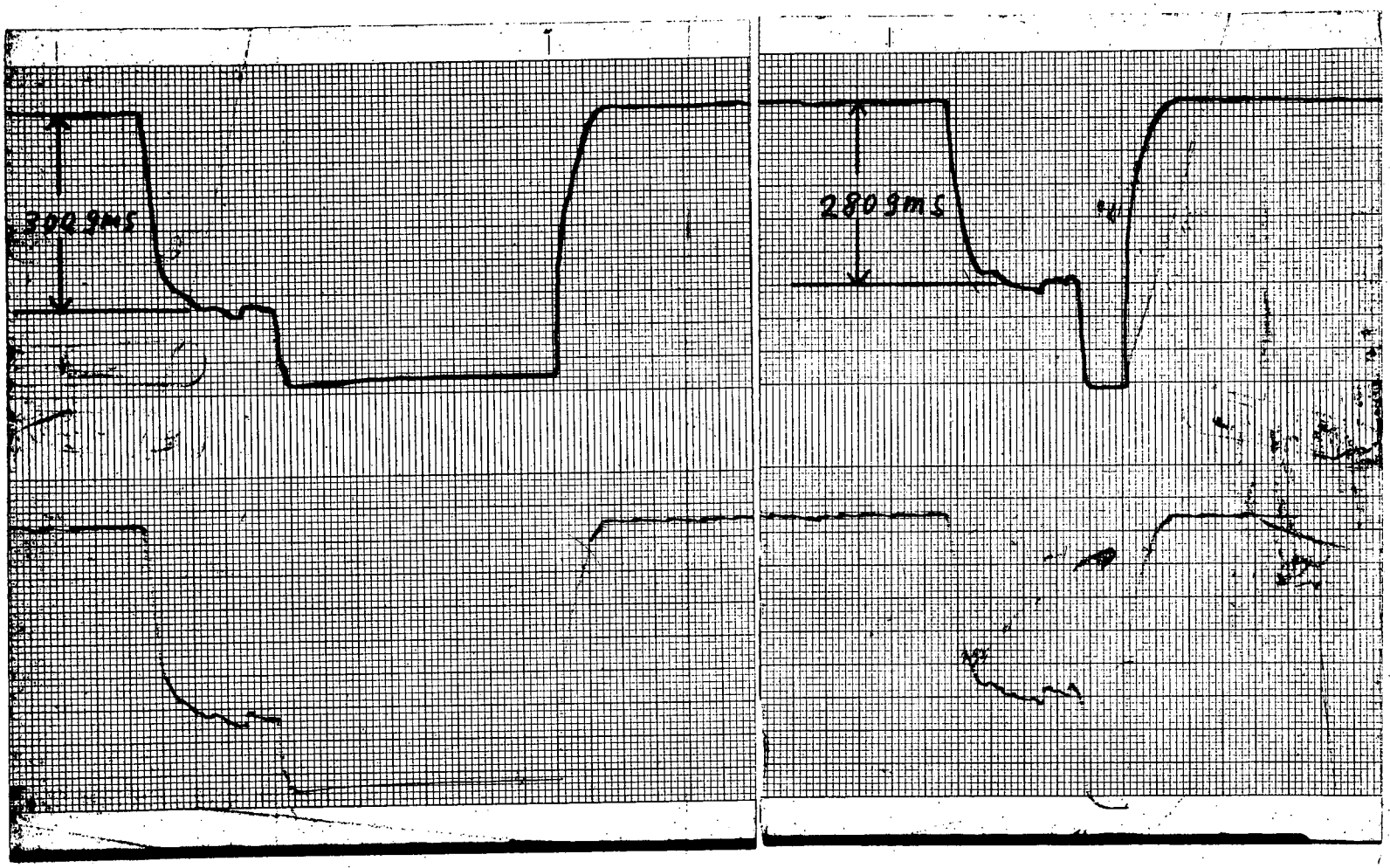


FIGURE 3

Mechanical Strength Test

Positive Plaques Experiment No. 6A

is a sharp increase in the load associated with the leveling points in the recorder tracings for Experiment 6A. This is the result of the movable portion of the Bend Tester fixture coming in contact with the stationary portion (see Figure 1). Experiment 6A test samples bent further into the "U" shape before achieving structural failure than Experiment 5A and almost exceeded the movable limit of the test fixture.

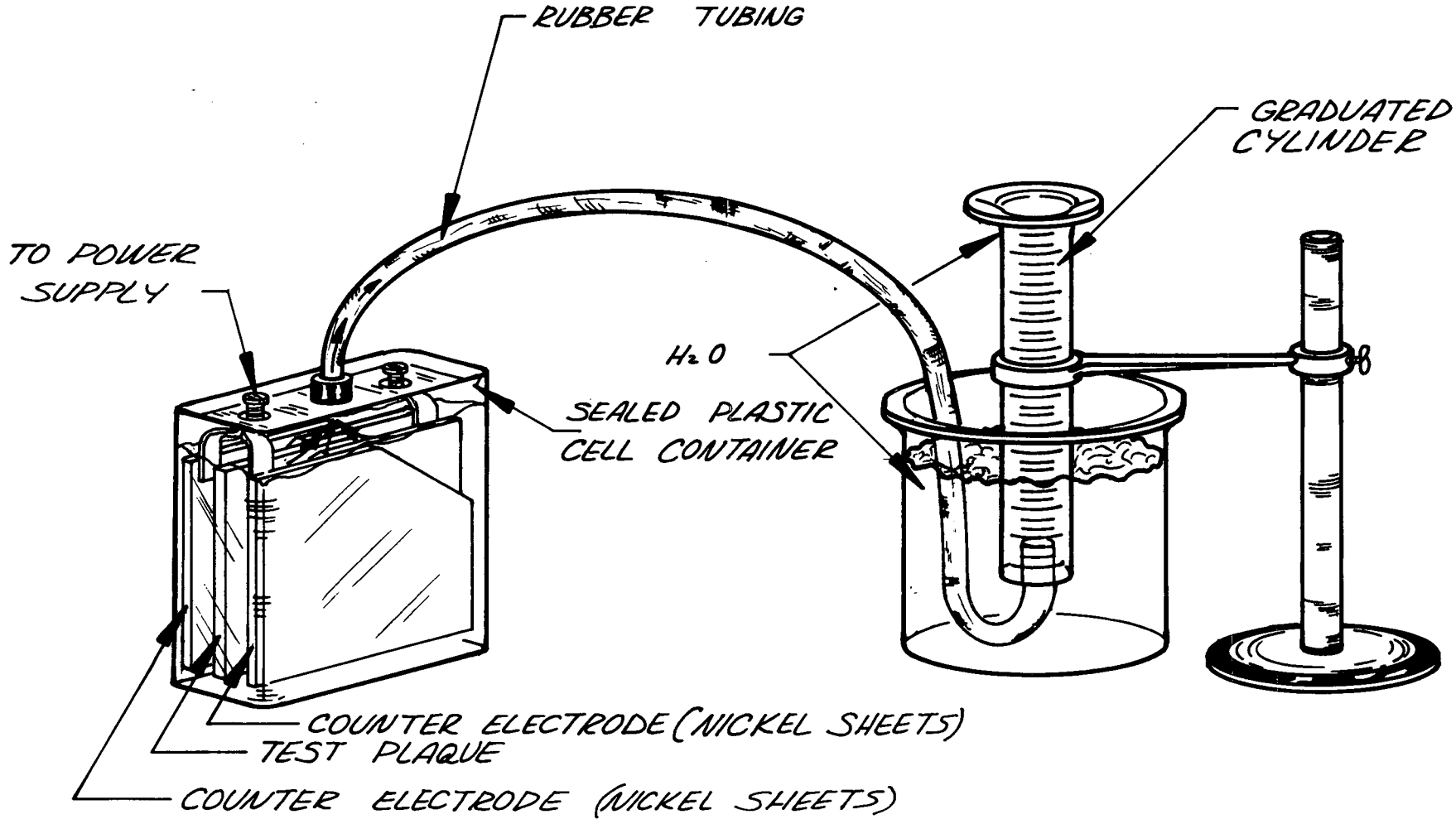
It would appear that the impregnation of positive plaques in nitrate solutions of high free acid levels does result in attack on the nickel matrix and subsequent loss of mechanical strength. But, in this case, it was not excessive and amounted to approximately a 10% loss. The impregnated plaques were, in any case, significantly stronger than the corresponding unimpregnated plaques. Tests performed on samples of the unimpregnated plaque lot used in the above experiment resulted in structural failure values in the order of 200 grams.

C. Polarizing Agent Study

It was observed during the previous impregnation/polarization experiments that polarization (conversion of impregnated material within the sintered nickel plaque to the active material form) in a KOH caustic solution resulted in a much more destructive effect upon the impregnated plaques (flaking of sintered nickel and active material, see Figures 2 and 3 of the Fourth Quarterly Report[1]) than polarization in a NaOH caustic solution. This phenomenon occurred under similar process conditions which included identical polarization current rates, identical solution specific gravities, identical polarization times and identical solution temperatures.

The only force associated with polarization process which it is believed could effect the observed plaque destruction, is the evolution of gas during the conversion reaction. To characterize the gas evolved between the two processes, an experiment was devised to measure the volume and the rate of gas evolved with caustic media as the single variable.

The physical test setup used in the experiment is shown in Figure 4. The procedure involved the identical impregnation of two plates (2 2.6" x 2.6" x 0.030" plates were immersed in a solution of nickel nitrate for one hour) which were then used as test standards. In the first half of the experiment, one of the impregnated plates was placed in the sealed plastic container and a solution of NaOH caustic was introduced. A four ampere polarizing current was passed for ten minutes; the gas evolved then passed through the connecting tube and displaced a certain volume of water in the graduated cylinder.



SYSTEM FOR MEASURING GAS EVOLVED DURING POLARIZATION

FIGURE 4

The following results were recorded for the experiment:

VOLUME OF WATER DISPLACED

TIME (MINUTES)	EXPERIMENT #1 (NaOH)	EXPERIMENT #2 (KOH)
0	0 ml	0 ml
5	50 ml	80 ml
10	130 ml	160 ml

It is apparent from the test results that a greater volume of gas was evolved during the experimental polarizing process in the case of a KOH caustic solution. Approximately 80 ml of gas was evolved in the first five minute interval as compared to 50 ml of gas for the NaOH caustic solution. During the second five minute interval, both experiments evolved equal volumes of gas (80 ml in both cases). It would appear that the mechanism evolved in differentiating the two caustic media occurs during the initial phase of the polarizing process.

It is assumed that the gas evolved affects plaque destruction by first being trapped in the recesses of the porous sintered nickel plaque, expanding by further evolution with a subsequent increase in pressure, and finally breaking up of the sintered nickel matrix to escape. The word "trapped" used in this explanation may be interpreted as referring to a situation in which gas cannot escape from an area at a rate great enough to support its evolution and thus resulting in increased pressure. With this assumption, an explanation is available to explain why polarization in KOH with the greater gas evolution results in a more destructive environment for the impregnated plaques.

A postulation of the exact mechanism which results in the different volumes of evolved gas must take into consideration that the same amount of current was passed in both experiments and would result in equal volumes of gas being evolved if dealing with the same gas mechanism. It is probable then two mechanisms of gas evolution are occurring concurrently at the impregnated plaque during the polarization process. First, the desired chemical conversion -

$$X(NO_3)_2 + H_2O + 8e^- \longrightarrow NH_3 + X(OH)_2 \quad (4) \qquad (1)$$

X = Ni or Cd

Equation Not Balanced

evolves a molecule of ammonia gas upon the transfer of eight electrons in the process of converting the impregnated material to the active material form. The second reaction which is assumed to be occurring is the electrolysis of H_2O.

$$2H_2O + 2\ e^- \longrightarrow H_2 + 2OH^- \quad (4) \qquad (2)$$

In this reaction, a molecule of hydrogen gas is evolved upon the transfer of two electrons.

By Avogadro's Law and the Kinetic Molecular Theory, it is known that different gases containing the same number of molecules, under the same temperature and pressure occupy the same volumes. It becomes obvious then the volume of gas evolved in the two processes in question depends upon the ratio in which the two reactions are occurring in each process while passing the same amount of current. If the first reaction predominated the passage of eight (8) electrons would result in the generation of one (1) molecule of gas. If, on the other hand, the second reaction predominated the passage of eight (8) electrons would result in the generation of four (4) molecules of gas.

To suggest why there may be difference in the ratio of these two (2) reactions occurring in the two (2) processes, it will be necessary to visualize a cross-section of an impregnated plaque. It is observed the impregnated material does not penetrate evenly into the sintered nickel matrix; the bulk of the material is located in the outer portion of the plaque (near the surface). Proceeding inward, the amount of impregnated material becomes smaller until it reaches its lowest concentration near the center of the plaque. It is further suggested the KOH caustic solution with its low viscosity (relative to the NaOH solution) and unusual penetrating ability (creeping effect) readily penetrates the inner areas of the impregnated plaque.

During the initial phases of polarization in a caustic solution of KOH, reaction of equation No.(1) predominates in the outer portion of the plaque matrix; but deeper into the plaque there is less impregnated material to support this reaction. The unavailability of impregnated material enhances reaction of equation No.(2) with its

much greater gas generation capacity. This phenomenon in effect occurs in the wrong place at the wrong time. The gas is generated deep in the nickel matrix where it is difficult to escape and the large $X(NO_3)_2$ molecules in the plaque pores have not been fully reduced to the smaller $X(OH)_2$ molecules which tends to further block the gas from escape.

It is assumed the NaOH caustic solution does not penetrate the nickel matrix as readily. The reaction of equation No. (1) predominates until the impregnation material is converted and until the caustic solution penetrates the inner recesses of the plaque matrix with the opening of the plaque pores as the results of the reaction of equation No. (1).

D. Spectrographic Analysis of Impregnation/Polarization Solutions

In the performance of the study of the process variables associated with the impregnation/polarization process, effort was expended in an evaluation of the chemical changes occurring in the various process solutions as they progressed through different stages of the operation. This investigation included the following:

(1) Determination of the normality of the caustic solution at the beginning and end of an experiment (wet titration method).

(2) Determination of the CO_3 content of the caustic solution at the beginning and end of an experiment (double end point, wet titration method).

(3) Determination of the NH_3 content of the caustic after the first cycle and the end of an experiment (distillation and titration).

(4) Free acid content (HNO_3) of the impregnation solutions at the beginning of each cycle (titration).

The data resulting from the above analyses was presented in the Appendix of the Fourth Quarterly Report.

In addition to these tests, the process solutions were subjected to a semi-quantitative spectrographic analysis for major impurities. In the following tables, the impurities detected are listed; the levels in which they are present are expressed in parts per million. In an example where the Experiment Number is preceded by "pre" or "post", this indicates that the sample for analysis was removed before performance of the experiment or after its completion. When no notation is involved, the test sample was removed before performance of the experiment. The letters "ND" means the element

was not detected during applicable test. The terms "major" and "minor" indicates the elements are major constituents of the test sample. Minor indicates the element which is lesser in concentration.

POSITIVE PROCESS IMPREGNATION SOLUTIONS, $Ni(NO_3)_2$

ELEMENTS DETECTED IN PPM

EXPERIMENT	Fe	Cu	Mg	Si	Co	Ca
#3	35	3.2	5.6	*	5000	500
#5	24	3.3	5.6	*	5000	500
#6	32	4.5	5.6	*	5000	500
#9	38	2.4	7.0	32	+5000	200
#10	15	2.4	7.2	35	+5000	200

*In these series of tests, information concerning detection of Si was not included in the laboratory analysis report.

NEGATIVE PROCESS IMPREGNATION SOLUTIONS, $Cd(NO_3)_2$

ELEMENTS DETECTED IN PPM

EXPERIMENT	Pb	Mg	Si	Fe	Al	Cu	Cd	Ag	Na	Zn	Ni	Co	Ca	Cr	K
#1	5	Minor	30	3	ND	20	Major	0.5	ND	ND	20	ND	9	ND	ND
#3	5	Minor	30	12	ND	15	Major	ND	ND	ND	Minor	ND	10	ND	ND
#4	5	Minor	30	20	ND	13	Major	ND	ND	ND	Minor	ND	7.2	ND	ND

POSITIVE PROCESS POLARIZATION SOLUTIONS

ELEMENTS DETECTED IN PPM

EXPERIMENTS	CAUSTIC	Si	B	Mg	Fe	Cu	Ni	Co	Ca	Al	Cr	K
Pre #5	NaOH	10	5	3	10	0.5	ND	ND	5	5	ND	100
Post #5		20	5	1	30	0.7	50	3	5	10	10	200
Pre #6	NaOH	10	5	5	10	0.7	3	ND	1	10	ND	100
Post #6		10	5	4	30	0.7	100	3	3	5	5	200
Pre #7	KOH	ND	ND	ND	ND	ND	ND	ND	1	ND	ND	Major
Pre #9	NaOH	10	*	5	20	0.5	5	ND	7	*	ND	*
Post #9		10	*	4	20	0.5	100	5	3	*	5	*
Pre #10	NaOH	10	*	1	20	0.5	50	3	5	*	5	*

*In these series of tests, information concerning the detection of the indicated elements was not included in the laboratory analysis report.

NEGATIVE PROCESS POLARIZATION SOLUTIONS, NaOH

ELEMENTS DETECTED IN PPM

EXPERIMENT	Pb	Mg	Si	Fe	Al	Cu	Cd	Ag	Na	Zn	Ni	Co	Ca	Cr	K
Pre #1	ND	1.1	15	ND	4	0.3	ND	8	Major	ND	ND	ND	5	ND	1500
Post #1	ND	0.3	10	ND	4	0.3	20	0.1	Major	ND	ND	ND	0.1	ND	500
Post #2	ND	1.3	10	3	7	0.3	100	ND	Major	ND	ND	ND	0.4	ND	1000
Pre #3	ND	2.6	8	1	4	0.3	ND	ND	Major	ND	ND	ND	1	ND	1000
Post #3	ND	0.5	16	3	6	0.3	30	ND	Major	ND	ND	ND	0.1	ND	1000
Pre #4	ND	0.3	8	ND	4	0.2	ND	0.1	Major	ND	ND	ND	0.6	ND	500
Post #4	ND	1.2	10	3	4	0.3	40	ND	Major	ND	ND	ND	0.3	ND	1000

Observations concerning the spectrographic analysis :

(a) Positive Process Impregnation Solutions

1. The high level of cobalt impurity is the result of an intentional impregnation solution additive.
2. The high level of calcium impurity cannot be explained, but it is common only to the positive impregnation solution indicating that it is an impurity in the raw material itself.

Conclusion - Repeated use of the positive impregnation solution within the limits of this work (10 experiments, 4 cycles each experiment) does not appear to result in the build-up of any particular impurity.

(b) Negative Process Impregnation Solutions

1. The high level of magnesium impurity is the result of an intentional impregnation solution additive.
2. Cadmium is indicated as a major constituent because the test sample is, of course, cadmium nitrate.
3. Nickel is a mild impurity initially, but after the first experiment and exposure of the impregnation solution to the sintered nickel plaques, the nickel impurity level increases considerably.

Conclusion - There appears to be a significant increase in the concentration of nickel with the repeated use of the negative impregnation solution. This accumulative build-up may become detrimental to the negative electrodes.

(c) Positive Process Polarization Solutions

1. As was the case with the negative impregnation solution, there is an increase in the nickel concentration associated with repeated use of the caustic solution during an experiment. The concentration level increase is considerably less than that associated with the impregnation solution.
2. Potassium appears to be a mild impurity which increases with use of the caustic solution. This probably results from a small residue of KOH remaining in the caustic storage tank (from previous use of KOH caustic solutions) and also a small residue in the impregnation/ polarization tank.
3. It is readily observed the purity level of the KOH caustic solution is superior to the NaOH solution. Calcium was the only impurity detected in the KOH solution.

Conclusion - There is a build-up in the concentration of nickel and potassium with repeated use of the caustic solutions, but it is doubtful either would be detrimental to the final positive electrodes.

(d) Negative Process Polarization Solutions

1. In the negative process, the build-up in nickel is replaced with a build-up of cadmium. This would indicate the nickel concentration increase associ-

ated with the positive polarization solution is not the result of the solubility of the sintered nickel as was the case in the impregnation solutions. Nickel was not detected in any negative caustic solution. The mechanism for impurity build-up in the polarizing solution must be the slight solubility of the impregnated material $Cd(NO_3)_2$ in one case and $Ni(NO_3)_2$ in the other.

2. Potassium is also a significant impurity in the negative caustic solutions, but there does not appear to be a pattern in its concentration level such as a buildup with use. This may be associated with error in the analysis or unrepresentative samples removed for tests.

Conclusion - There is a build-up in the concentration of cadmium with repeated use of the caustic solutions and high concentration levels of potassium also exist; but, again it is probable that neither of these impurities would be detrimental to the final negative electrodes.

III. SEPARATOR MATERIAL STUDY

The "Process Variable Study" Program has been primarily concerned, up to this point, with the study of process variables associated with the raw plaque laying and sintering process, and the sintered plaque impregnation/polarization operation. The program is, however, intended to study all major phases of the nickel-cadmium cell manufacturing process. During this reporting period, a new phase was introduced with the study of the characteristics of various types of separator materials.

The type of tests to be used in this characterization process are defined by Eagle-Picher Industries' Document EP-MS-118, "Non-Woven Nylon Specification for Nickel-Cadmium Spacecraft Cells" (see Third Quarterly Report[2]). This document reflects the specific nickel-cadmium cell separator tests described in the "Interim Model Specification for High Reliability Nickel-Cadmium Spacecraft Cells", prepared by NASA/ Industry Committee, April 30, 1969. In addition, the present effort will include a visual characterization of the various separator materials by scanning electron photomicrographic examination.

The separator material study is intended to serve a dual purpose. First, the subject separator materials will be characterized as to their applicability for use in nickel-cadmium power systems. Second, the characterization tests themselves will be evaluated to determine if they evolve useful information and merit retention in nickel-cadmium, aerospace separator material specifications.

Within the time limits of the present reporting period, the selected separator materials were subjected to only three (3) of the proposed characterization tests which were as follows:

(a) Scanning electron photomicrographic examination for physical attributes.

(b) Methanol extraction for soluble organic content with subsequent IR analysis of extract for major organic constituents.

(c) Qualitative and quantitative chemical analysis for inorganic content.

The original scope of work included two (2) separator materials for study. These were Pellon 2505, a non-woven nylon material supplied by the Pellon Corporation and "Lyonel" also a non-woven nylon material supplied by Howard Textile Mills, Inc. However, at the time of performance of the above tests, several other types of separator material samples of interest were available and they were included in this initial series of characterization tests. The following is a list of the separator materials included in the present work:

Non-Woven Nylon

1. Pellon 2505; supplied by Pellon Corporation, 221 Jackson Street, Lowell, Massachusetts 01852.
2. "Lyonel" (unit weight 2 ounces/sq.yard), supplied by Howard Textile Mills, Inc., 65 West 36th Street, New York, New York 10018.

Non-Woven Polypropylene

3. FT-2140, supplied by Pellon Corporation.
4. WEX-1242, supplied by GAF Corporation, Industrial Products Division, Glenville Station, Greenwich, Connecticut 06830.

C-2

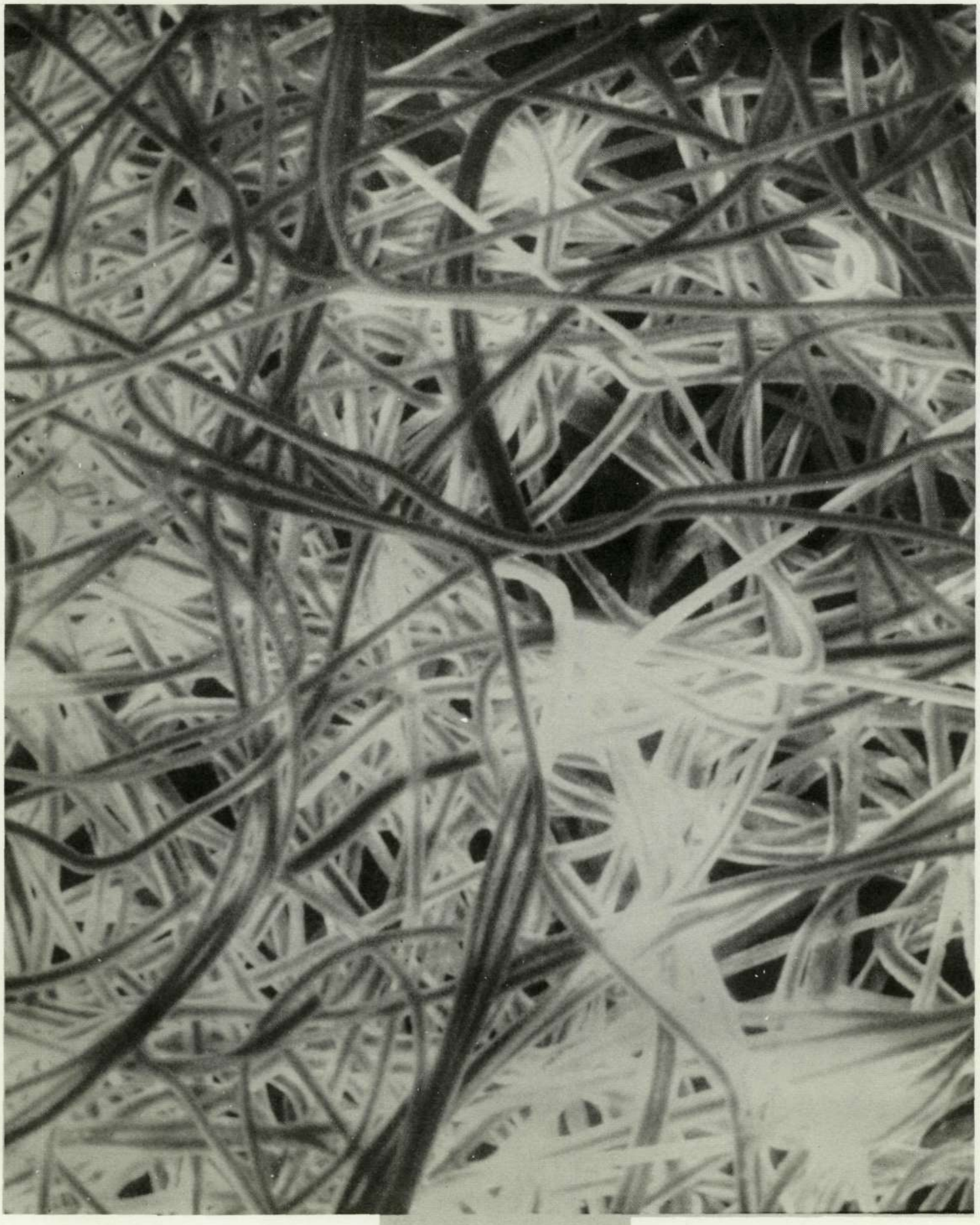
Special Polypropylene

5. "100% Woven Cloth Polypropylene", supplied by Howard Textile Mills, Inc."

6. "Microweb" Micropolypropylene, supplied by Hercules Incorporated, Fiber and Film Department, P. O. Box 12145, Research Triangle Park, North Carolina 27709.

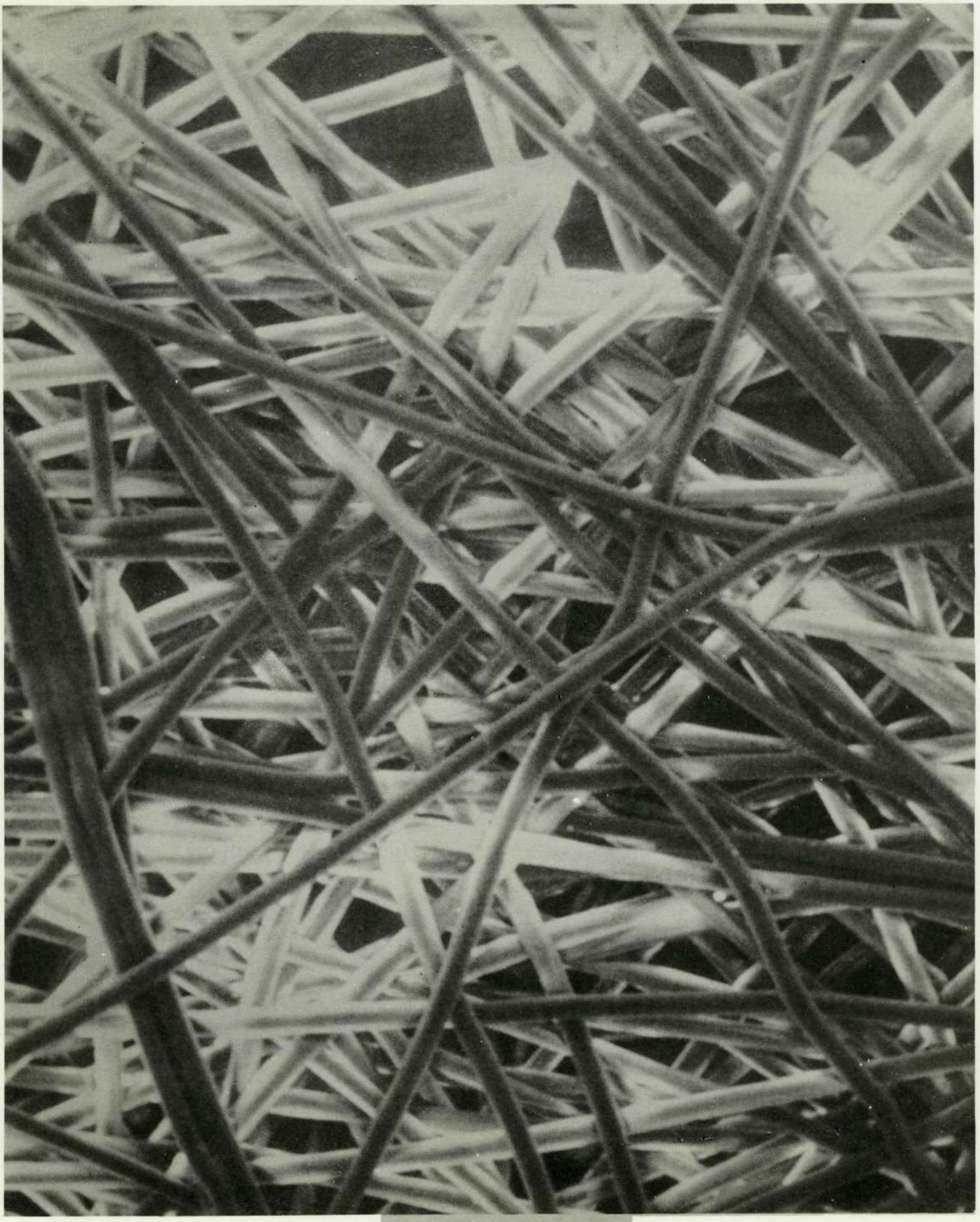
A. Scanning Electron Photomicrographic Examination

Samples of the selected separator materials were supplied to Rocky Mountain Technology, Inc. located in Golden, Colorado for performance of the scanning electron photomicrographic examination. The following photomicrographs are the results of this effort:

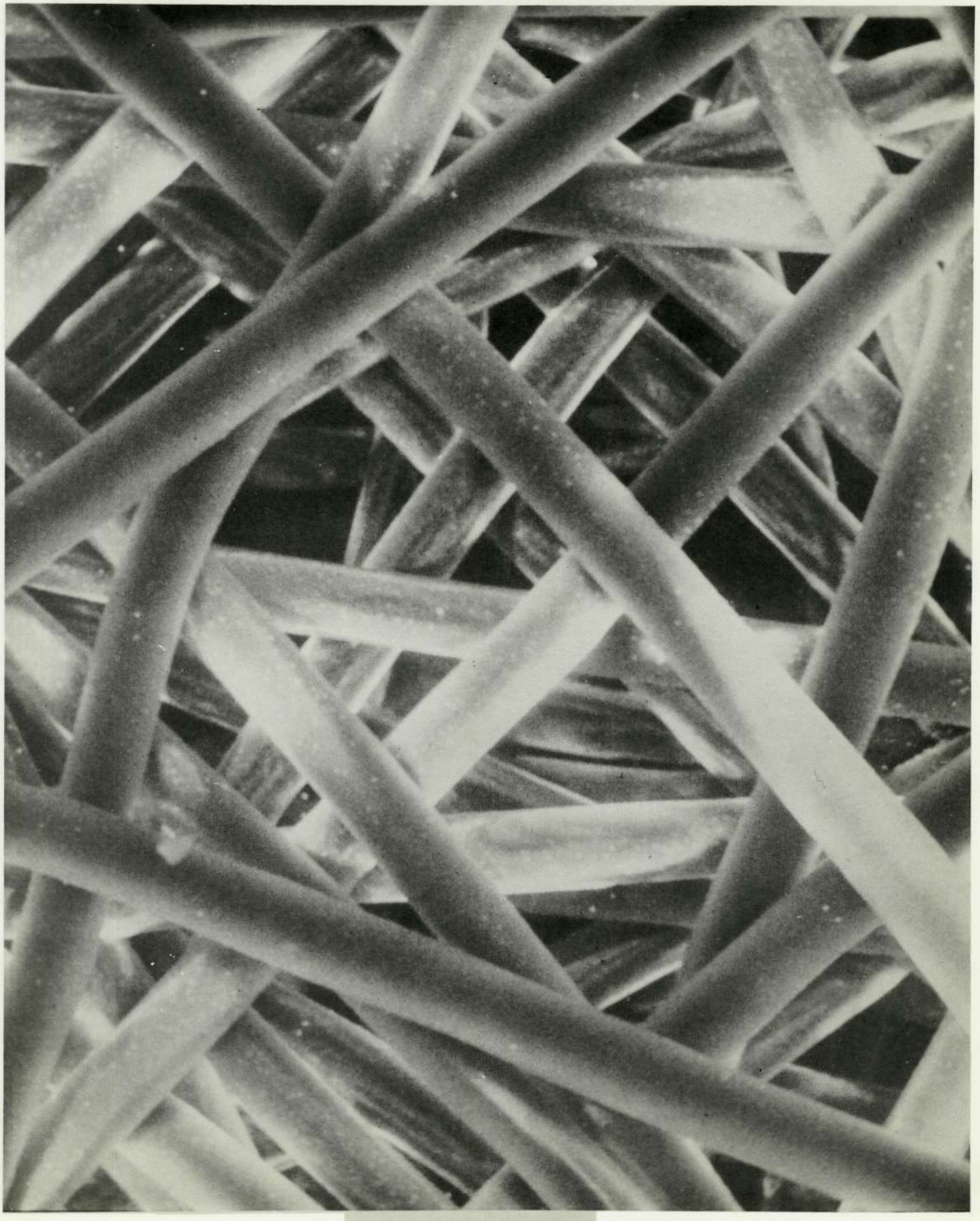


MICROGRAPH 19
PELLON 2505
250X Magnification

MICROGRAPH 20
PELLON 2505
750X Magnification

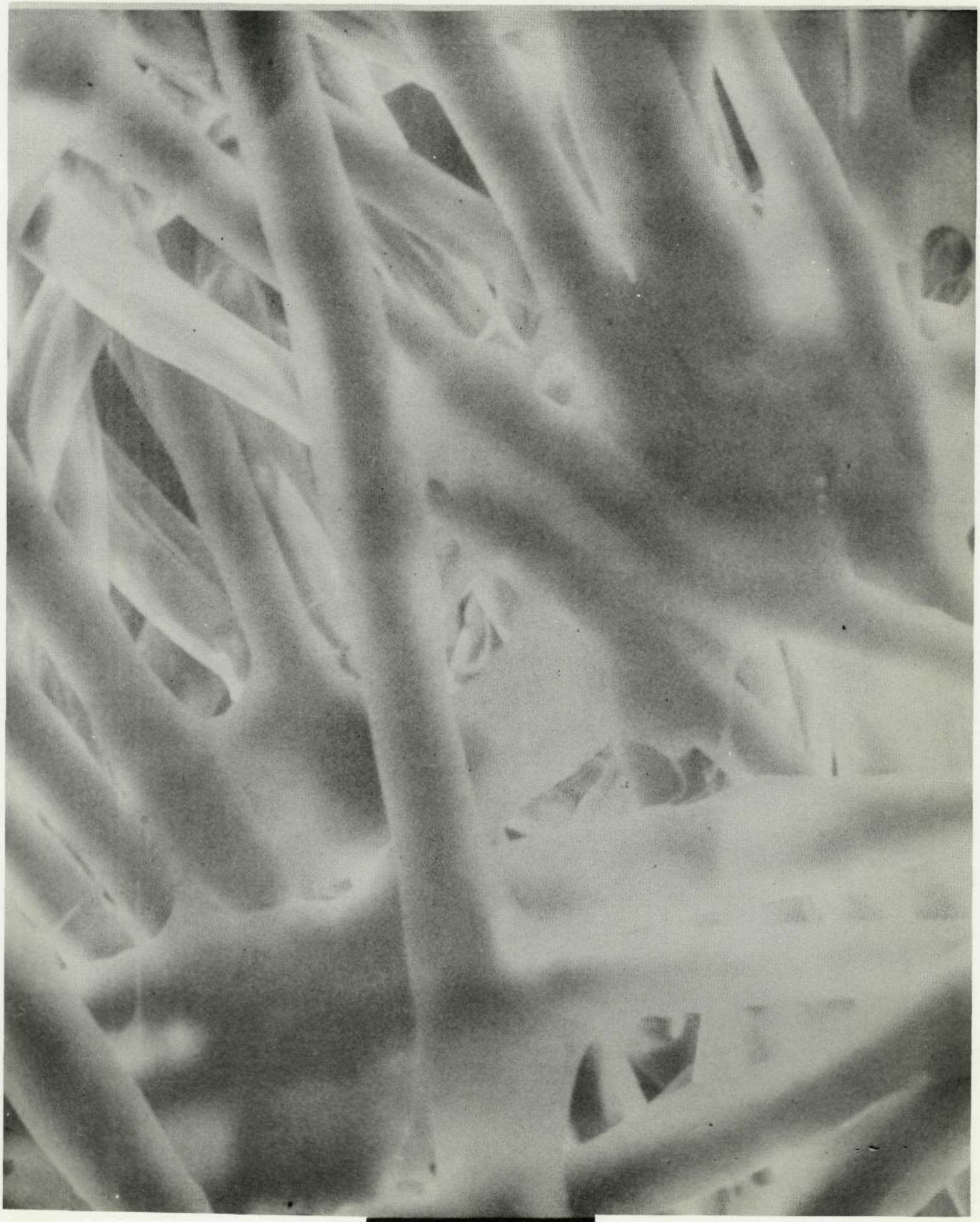


MICROGRAPH 21
"LYONEL"
250X Magnification



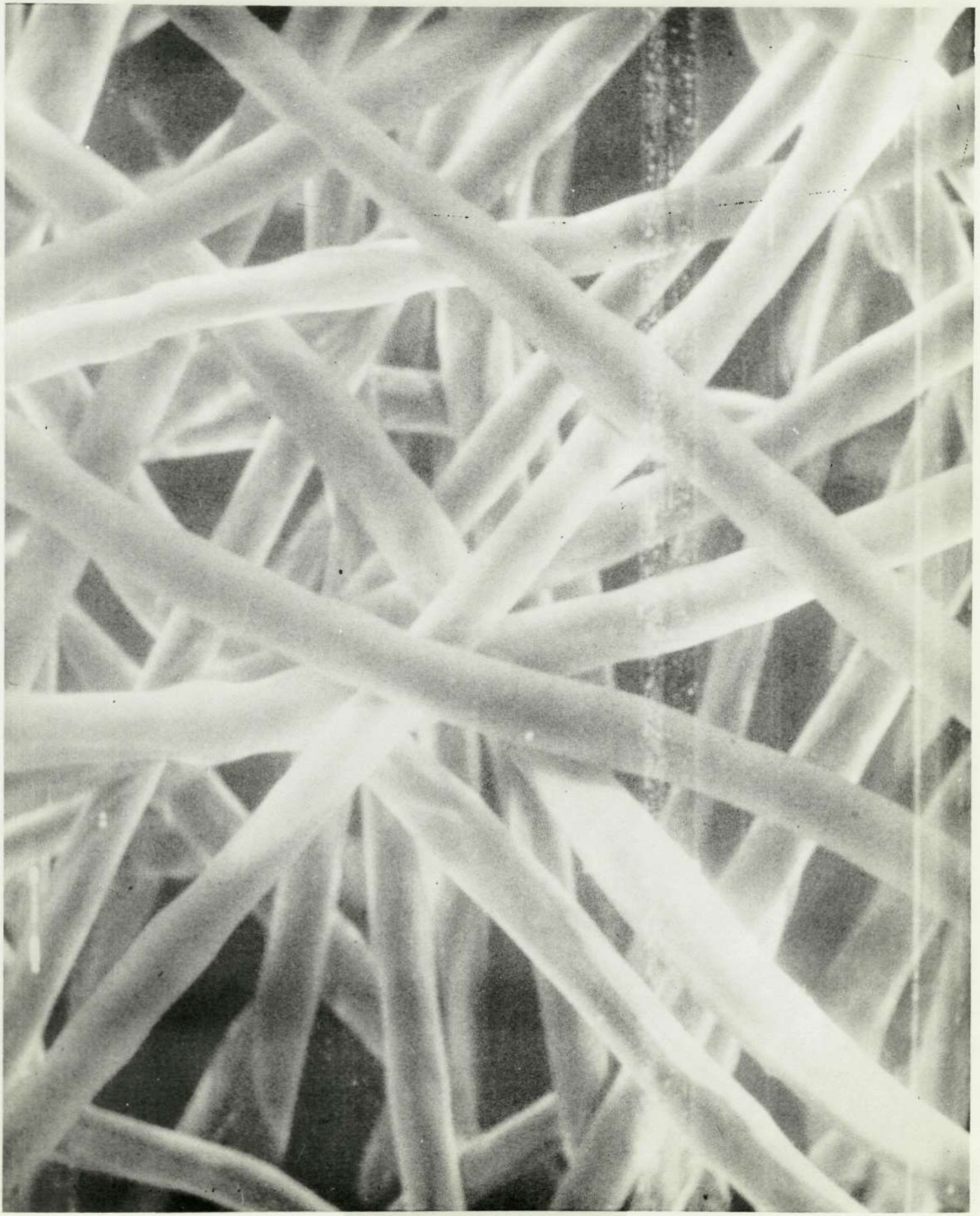
MICROGRAPH 22
"LYONEL"
750X Magnification

MICROGRAPH 23
FT-2140
250X Magnification

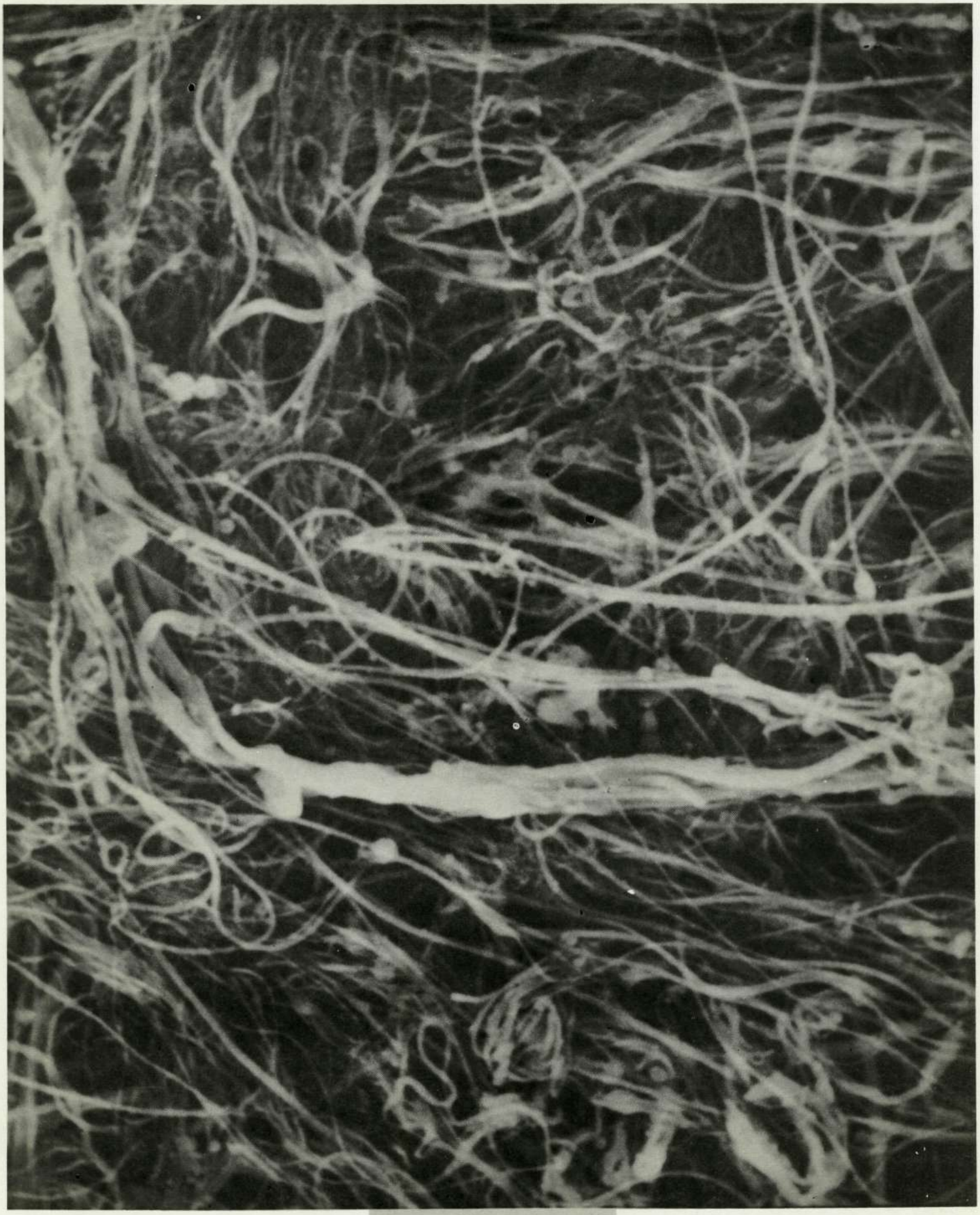


MICROGRAPH 24
FT-2140
750X Magnification

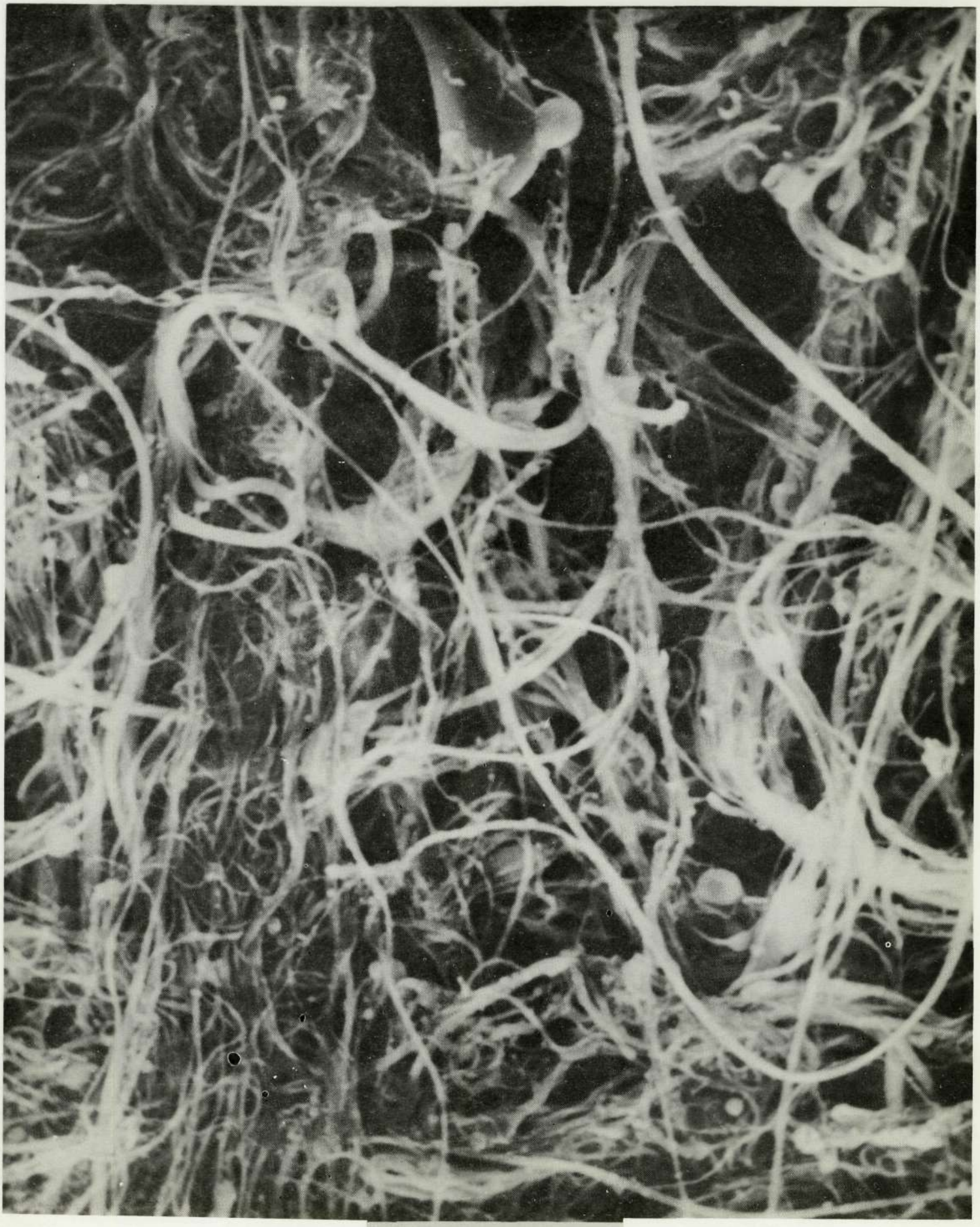
MICROGRAPH 25
WEX-1242
250X Magnification



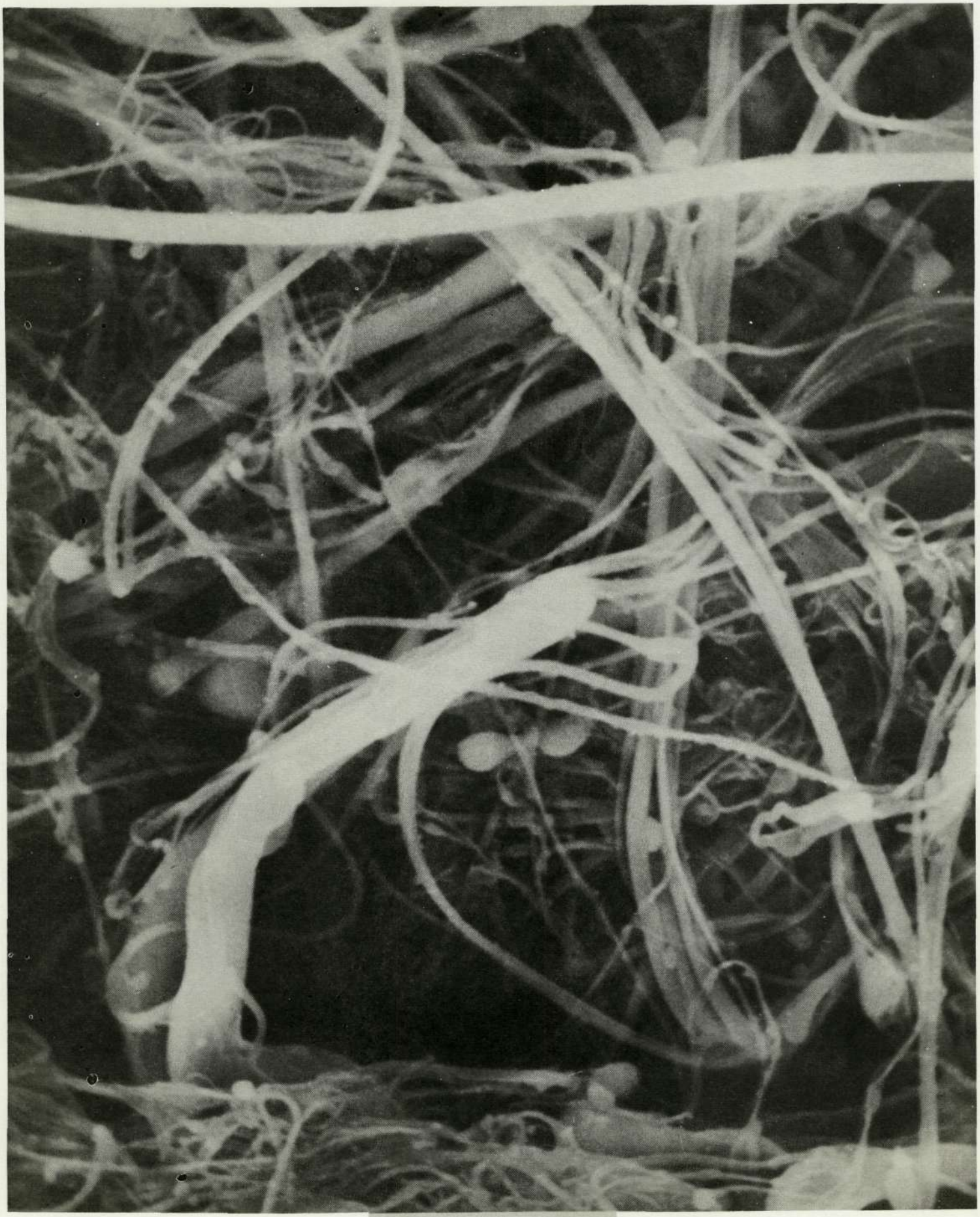
MICROGRAPH 26
WEX-1242
750X Magnification



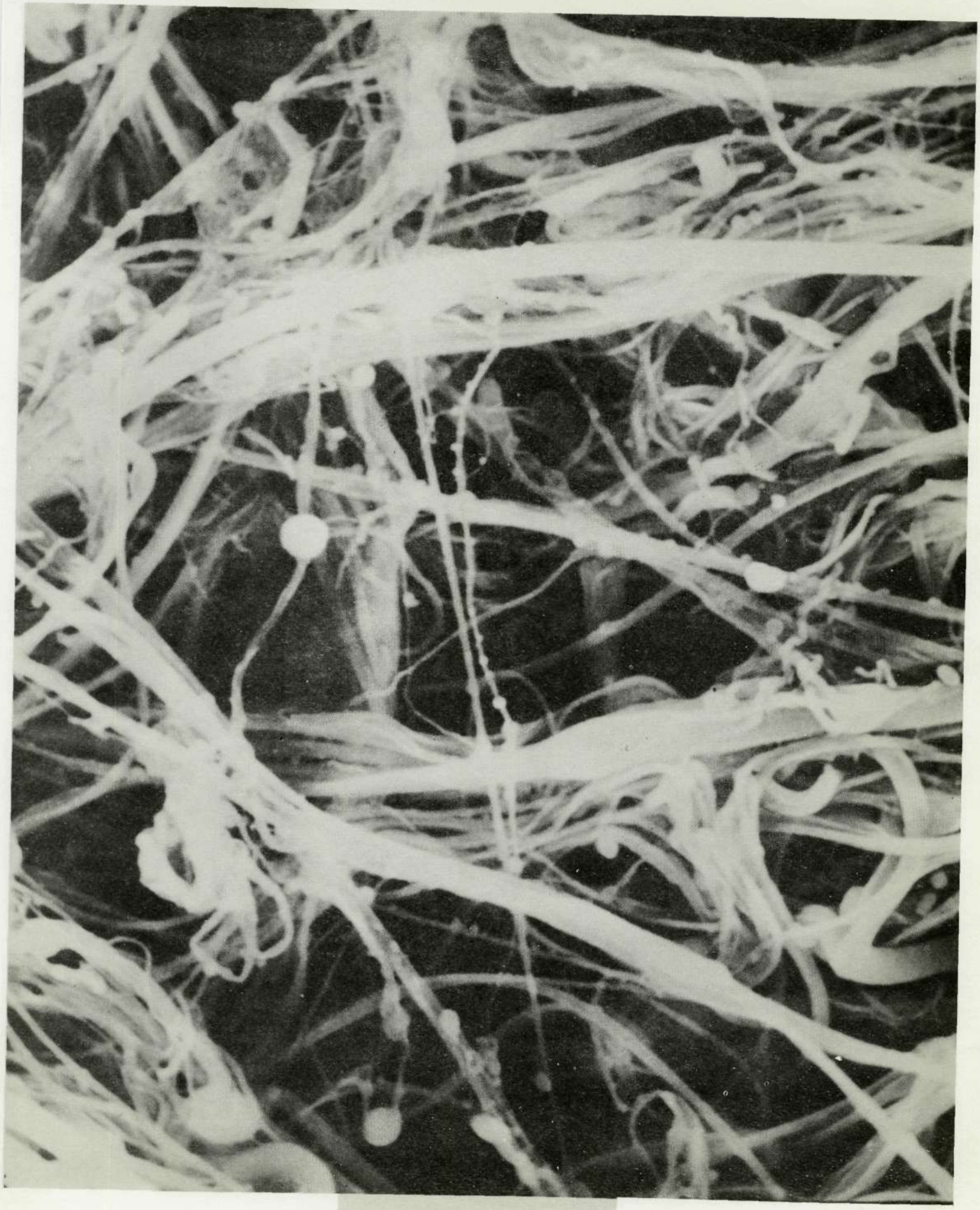
MICROGRAPH 27
"MICROWEB"
625X Magnification



MICROGRAPH 28
"MICROWEB"
625X Magnification



MICROGRAPH 29
"MICROWEB"
1250X Magnification



MICROGRAPH 30
"MICROWEB"
1250X Magnification

Observations concerning the photomicrographic study of separator materials:

1. Individual filament size (diameter) and distribution appear to be both similar and uniform in both the nylon and polypropylene separator materials. The obvious exception is the "Microweb" separator material. In this case, the individual filaments vary considerably in size and are poorly distributed through the material (filaments tend to form aggregates leaving open areas).
2. Some form of residue or contamination appears to be associated with the filaments of the Pellon 2505 separator material (Note Micrograph 20). This is observed as somewhat of a ragged edge on a particular filament.
3. Fusion of filaments if observed in the FT-2140 and "Microweb" materials (Note Micrographs 24 and 30). This phenomenon occurs to a lesser extent in the WEX-1242 material (Note Micrograph 26).
4. The "Lyonel" separator material (Note Micrographs 21 and 22) distinguishes itself by exhibiting not only uniform filament diameters but also a uniform diameter through the length of an individual filament. In addition, it exhibits the cleaner appearing filaments.

B. Extractable Organic Content and IR Analysis

In the following procedure, the extractable organic content is defined as that portion of a separator material which is soluble in methanol.

The experiment proceeded with the selection of approximately a 10 cm squared piece of separator from each material under investigation with exception of the micropolypropylene which was

not yet available. Each sample was analytically weighed and then immersed in a beaker containing 100 ml of anhydrous methanol. The sample was agitated mechanically for 30 minutes and then the procedure repeated for a second and third beaker each containing fresh methanol. After completion of this process, the samples were carefully dried and reweighed. The difference in the starting and end weight is expressed as the percent weight loss or the percent extractable organic content.

The following is a summation of the results of this experiment:

TABLE I

Separator Material as Received from Vendor

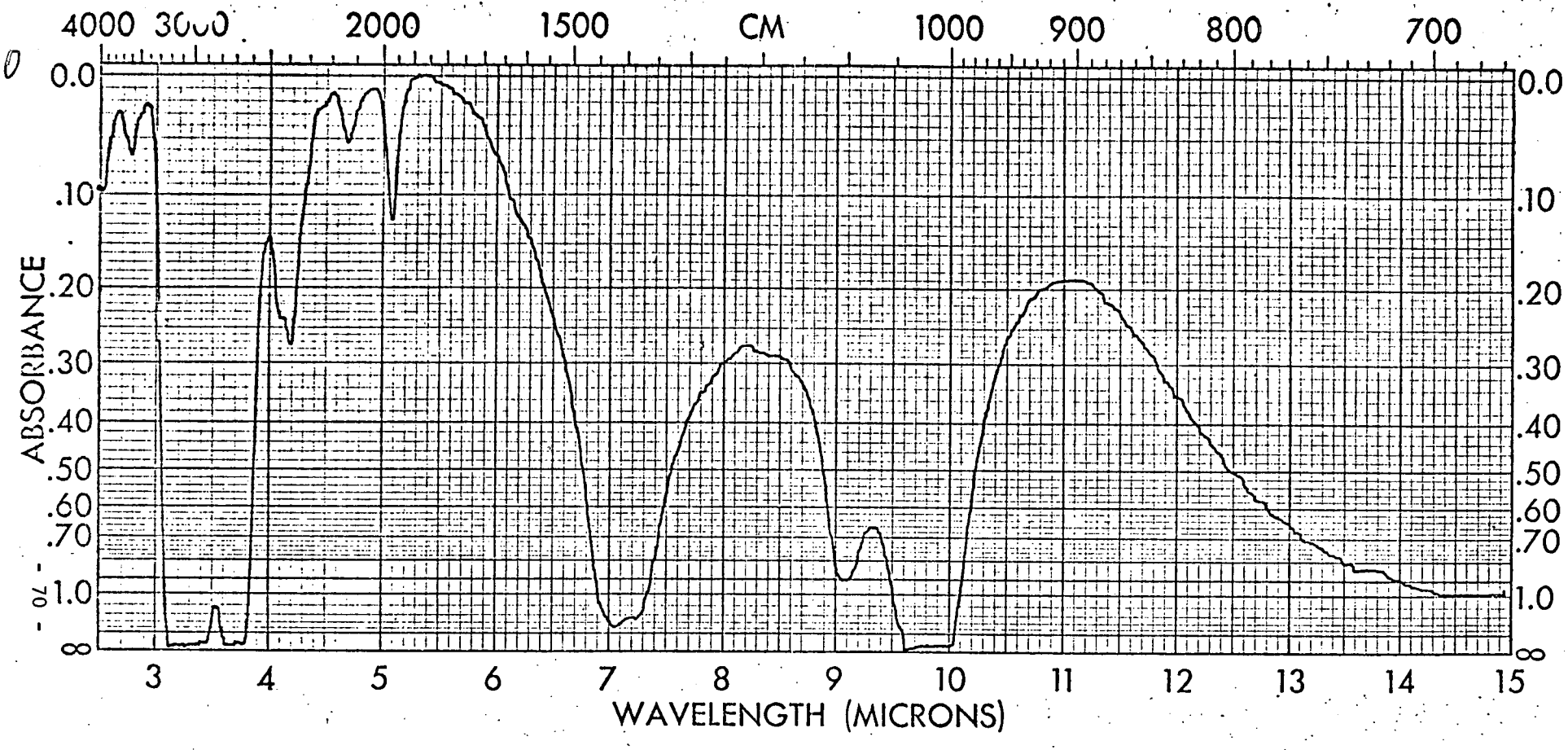
Material Sample	#1 Pellon 2505	#2 "Lyonel"	#3 FT-2140	#4 WEX-1242	#5 Woven Cloth
Wt. Start (Grams)	0.7039	0.8118	0.7911	0.8368	0.4899
Final Wt.	0.6883	0.7943	0.7871	0.8330	0.4864
Wt. Loss	0.0136	0.0175	0.0040	0.0038	0.0035
% Extract	1.932	2.156	0.506	0.454	0.714

The "Lyonel" separator material demonstrated the greater percent of extractable organic material. This is followed closely by the second nylon separator material, Pellon 2505. The soluble contaminant is probably a wetting agent or possibly a residue from some treatment necessary to the nylon separator manufacturing process. The polypropylene separator materials demonstrated significantly less extractable quantities. It would appear the use of polypropylene materials may result in less contamination of the cell system from soluble organic materials.

An attempt was made to identify the major organic constituents of the extraction by IR analysis. An aliquot of the methanol residue from the first wash beaker for separator material sample No. 3, FT-2140, was furnished to an independent laboratory (Bruce Williams Laboratories, Joplin, Mo.) for an IR analysis. The results of this first attempt were not satisfactory (see Figure 5). They reported it was not possible to determine the major organic constituents in the methanol extract, as the effects of the water of hydration associated with methanol masked the desired absorption bands.

The laboratory was then supplied a sample of the FT-2140 separator material and they performed their own extraction using carbon tetrachloride as a solvent. An IR analysis on this extract rendered distinct absorption bands which could be used for the purpose of identifying the major organic contaminants (see Figure 6). The organic contaminants were reported to be similar to a fatty acid such as soybean oil and linseed oil with a small amount of amine salts.

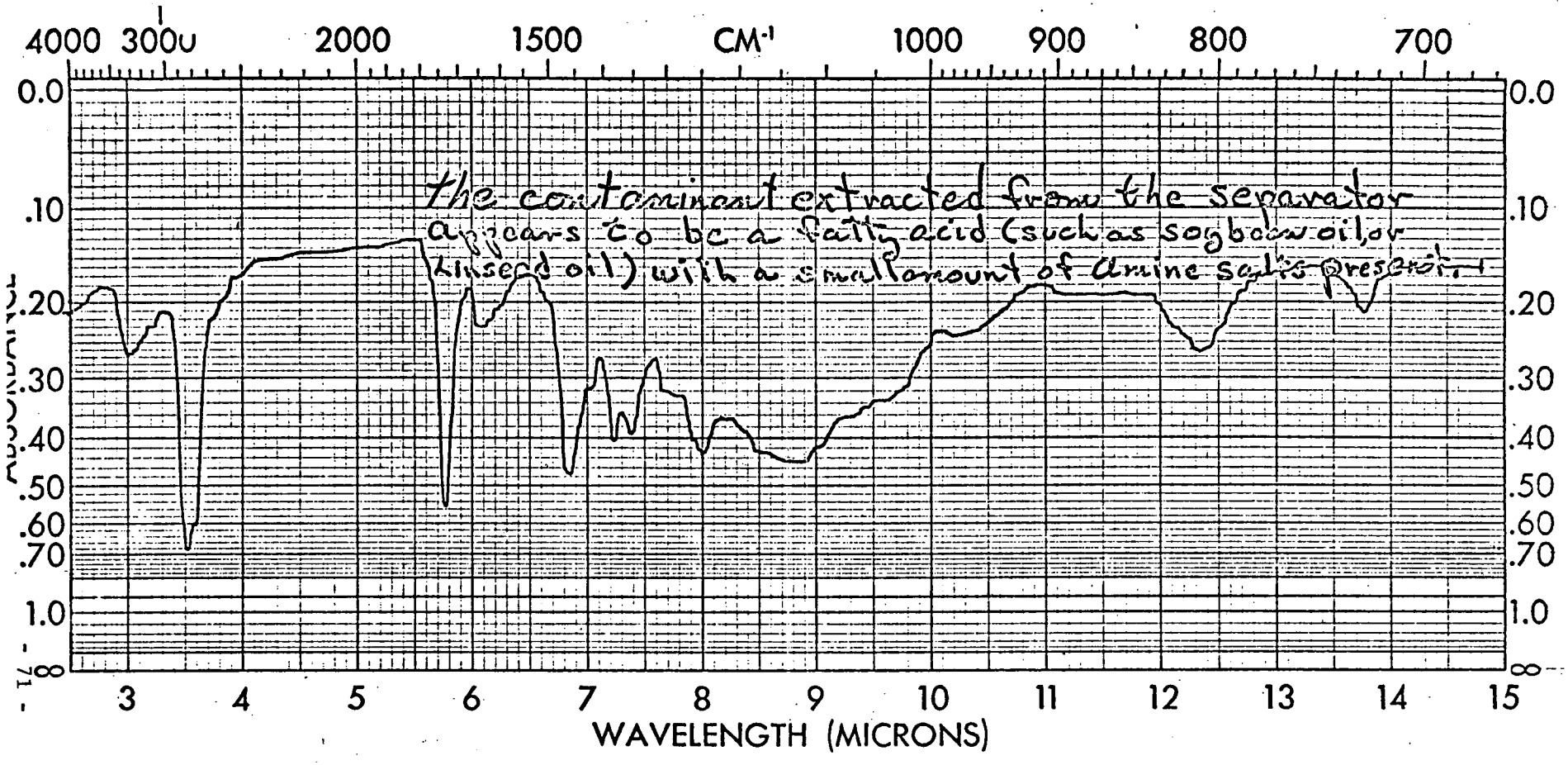
In this particular case, the carbon tetrachloride extraction appeared to be a superior means of identifying the soluble organic constituents associated with the separator materials. However, tests would need to be performed to determine if this solvent attacks the separator material itself before it could be adopted for the total soluble organic test procedure described above.



SPECTRUM NO. 706	ORIGIN Eagle Picher Industries, Inc. Couples plant Joplin, Mo.	LEGEND	REMARKS The Contaminant is unidentifiable in the Methanol extract.
SAMPLE #940193 1-A (Methanol extract)	PURITY	1.	
	PHASE Liquid	2.	
	THICKNESS .025 mm.	DATE 15 April 71	
		OPERATOR G. Lee	

SPECTRUM NO. _____ SAMPLE _____

FIGURE 5



SPECTRUM NO. 741	ORIGIN Eagle Picher Industries, Inc. Couples Plant Joplin, Mo.	LEGEND	REMARKS the separator was extracted and solvent evaporated from a sodium chloride plate leaving a film of the contaminent.
SAMPLE #940424	PURITY	1.	SPECTRUM NO.
	PHASE Liquid	2.	SAMPLE
	THICKNESS Film	DATE 6 May 71	
		OPERATOR G. Lee	

FIGURE 6

Other investigators have reported success using the methanol extraction procedure. It may be that in the above extraction, too great a volume of methanol was used for the subject sample size, resulting in a dilution masking effect of the organic constituents.

The evaluation of this test will be continued with a number of the remaining separator materials and should render available additional information for comparison.

C. Inorganic Content Analysis

Three (3) approaches were undertaken to characterize the inorganic content of the various separator materials. The first approach involved a wet chemical analysis on extract aliquots for the following contaminants: nitrate, silica, carbonate, nickel, zinc and chlorine. Samples of separator material were placed in beakers containing 50 ml of distilled water and magnetically stirred for 16-24 hours. Aliquots for chemical analysis were then removed.

The second approach involved a complete ignition of the separator material and the analytical weighing of the remaining ash. The ash is assumed to consist of the inorganic impurities.

The third and final approach involved a semi-quantitative spectrographic analysis of the ash resulting from the above procedure.

In addition to performance of tests on separator materials as received from the vendors, samples of the separator materials were washed in methanol (see extraction procedure above) and the subject tests performed on the washed samples.

The following tables summarize the data evolved from these tests.

TABLE NUMBER II

WET CHEMICAL ANALYSIS
(PPM)

SAMPLE	CHLORINE	NI*	ZN*	NO_3	SIO_2	CO_3
#1 Pellon 2505						
As Received	Trace	.06	40.00	ND	ND	ND
Washed	Trace	.05	2.10	ND	ND	ND
#2 "Lyonel"						
As Received	ND	.05	.24	ND	ND	ND
Washed	ND	.05	.22	ND	ND	ND
#3 FT-2140						
As Received	ND	.05	.16	ND	ND	ND
Washed	ND	.05	.10	ND	ND	ND
#4 WEX-1242						
As Received	ND	.11	.18	ND	ND	ND
Washed	ND	.07	.13	ND	ND	ND
#5 Woven Cloth						
As Received	ND	.20	.10	ND	ND	ND
Washed	ND	.05	.16	ND	ND	ND

*Atomic Absorption Method

TABLE NUMBER III

IGNITION ANALYSIS

SAMPLE	PERCENT OF STARTING WEIGHT
#1 Pellon 2505	
As Received	0.418
Washed	0.026
#2 "Lyonel"	
As Received	Trace
Washed	Trace
#3 FT-2140	
As Received	0.012
Washed	Trace
#4 WEX-1242	
As Received	Trace
Washed	Trace
#5 Woven Cloth	
As Received	Trace
Washed	0.849

TABLE NUMBER IV

SEMI-QUANTITATIVE SPECTROGRAPHIC ANALYSIS
(PPM)

SAMPLE	Cs	B	Mg	Si	Fe	Cu	As	Zn	Ti	Ca
#1 Pellon 2505										
As Received	ND	10	3	50	3	1	2	1000	500	30
Washed	ND	ND	3	20	3	3	5	ND	200	10
#2 "Lyonel"										
As Received	ND	5	3	20	5	10	2	ND	ND	10
Washed	100	ND	5	50	3	3	3	ND	ND	10
#3 FT-2140										
As Received	ND	ND	1	50	ND	ND	1	ND	ND	10
Washed	ND	ND	3	100	3	1	2	ND	100	50
#4 WEX-1242										
As Received	ND	ND	.5	20	ND	.5	1	ND	ND	2
Washed	ND	ND	5	ND	ND	ND	.5	ND	ND	ND
#5 Woven Cloth										
As Received	ND	ND	1	20	ND	.5	2	ND	500	30
Washed	ND	ND	.5	10	ND	.5	1	ND	50	2

Reviewing the analytical data presented in the three (3) tables, the inorganic impurities detected generally appear to be minor and randomly distributed through the various separator materials. However, a high incidence of Zn impurity is associated with Pellon 2505 (Sample #1) in the "as received" state. This is evidenced in both the wet chemical analysis and spectrographic data. In addition, somewhat higher levels of Ti are associated with Pellon 2505 (Sample No. 1) and 100% woven cloth polypropylene (Sample No. 5) materials.

The methanol wash procedure does not appear, in general, to be effective in reducing the inorganic impurity levels. However, the Zn impurity level associated with Pellon 2505 (Sample No. 1) is an exception. The impurity level is significantly reduced by the treatment as evidenced again in both the wet chemical analysis and spectrographic data. There is also some evidence of a reduction in the Ti impurities.

The amount of ash remaining in the ignition analysis appears to be minor for the samples tested. A peculiar result was encountered with the woven cloth (Sample No. 5); the amount of ash residue increased for some unexplained reason with the methanol washed sample.

D. Separator Study Summation

Summarizing the effort in the separator material study during this reporting period, the three (3) tests utilized during this period (photomicrographic, organic and inorganic analysis) appears to offer some useful information in the characterization of various separator materials from an R & D standpoint. However, correlations between the various parameters measured and the effect upon the function of the separator material in nickel-cadmium systems would

have to be identified to justify the incorporation of all the tests in a nickel-cadmium separator material specification.

From a quality assurance standpoint, a number of the analytical tests merit consideration. A standard separator material quality level could be maintained by specifying specific organic extraction, inorganic ignition, and semi-quantitative spectrographic requirements.

IV. CONCLUSIONS AND RECOMMENDATIONS

1. A scanning electron photomicrographic examination of impregnated positive and negative plaques resulted in the following observations:

   a) Positive and negative plaques are distinguished by the entirely different appearance of the impregnated active materials. The positive material presents almost a continuous mass (broken up by surface cracks) with little evidence of discrete or crystal shaped particles. The negative material is just the opposite being made up of distinct crystal shaped particles.

   b) The vacuum impregnation of positive plaques under a condition of low nitrate solution temperatures (150°F) results in impregnated plaques exhibiting very rough and porous surfaces.

   c) The impregnation (no vacuum) of positive plaques under a condition of high nitrate solution temperatures (200°F) results in impregnated plaques exhibiting very smooth and non-porous surfaces.

   d) Longer polarization times (one hour) used in the positive plaque impregnation process appears to result in the greater compaction of the active material into the sintered nickel matrix of the plaque.

   e) Longer polarization times (one hour) used in the negative plaque impregnation process appears to result in a significant reduction of the size of the crystal shaped particles.

2. The positive process impregnation of sintered nickel plaques in nitrate solutions containing high free acid levels (4 grams/liter of HNO_3) results in attack upon the nickel matrix and a subsequent loss of mechanical strength. The loss, however, appears to be small, amounting to only 10% of the mechanical strength of plaques impregnated in nitrate solutions containing low free acid levels (1 gram/liter).

3. The more destructive effect upon positive impregnated plaques from polarizing in a KOH caustic solution compared to a NaOH solution is believed the result of a larger volume of gas being generated deep in the sintered nickel matrix when using the KOH solution.

4. A semi-quantitative spectrographic analysis of impregnation/polarization process solutions resulted in the following conclusions:

   a) The repeated use of the positive process impregnation solution ($Ni(NO_3)_2$) did not result in any measurable build-up of a particular impurity.

   b) The repeated use of the positive process polarization solution (KOH or NaOH) resulted in the build-up of nickel and potassium impurity levels.

   c) The repeated use of negative process impregnation solution ($Cd(NO_3)_2$) resulted in an increase of the concentration of nickel.

   d) The repeated use of the negative process polarization solution (NaOH) resulted in the build-up of cadmium and potassium impurity levels.

5. Selected separator materials were subjected to a series of tests (scanning electron photomicrographic examination for physical

characteristics, methanol extraction for soluble organic content, and chemical analyses for inorganic content) for the purpose of characterizing the separator material and evaluating the particular tests for its applicability to separator material specifications. The following conclusions evolved from this process:

Photomicrographic Examination

a) The five (5) separator materials study (two nylons and three polypropylenes) generally demonstrated uniform filament size and distribution within a material and from separator to separator. However, the new Hercules "Microweb" micropolypropylene separator material presented a radical deviation from the norm with very un-uniform filament size and distribution.

b) The Pellon 2505 nylon separator material appeared to have some form of contamination associated with the individual filaments (evidenced by rough filaments surfaces).

Methanol Organic Extraction

c) The nylon separator materials studied demonstrated greater extractable organic contents than the polypropylene materials (typically 2.0% compared to 0.5% of sample weight).

d) In an attempt to determine the major organic constituents in the methanol extract by IR analysis, very poor results were obtained. An extraction was then performed in carbon tetrachloride with significantly improved results.

e) An IR analysis (using carbon tetrachloride as the extraction media) identified the major extractable organic constituents associated with the Pellon FT-2140 polypropylene separator as a material similar to a fatty acid such as soybean oil or linseed oil.

Inorganic Analysis

f) Significant quantities of Zn impurity (1000 ppm) was detected in the Pellon 2505 nylon separator material. In addition, quantities of Ti impurity (500 ppm) were detected in Pellon 2505 and the 100% woven cloth polypropylene material.

g) Generally, the washing of separator materials in methanol does not appear to effectively reduce the inorganic impurity levels. However, in the case of Pellon 2505, a significant reduction in the Zn impurity level was obtained by the washing procedure.

6. The various tests utilized in the separator material study during this reporting period appear to be useful for characterizing separator materials, but they are not recommended in their entirety for incorporation in cell separator material specification. Particular analytical tests (organic extraction, ignition analysis and spectrographic analysis) could, however, be recommended for quality assurance sections of the above documents to assure a standard quality separator material is obtained.

REFERENCES

1) Fourth Quarterly Report, Study of Process Variables Associated With Manufacturing Hermetically-sealed Nickel-Cadmium Cells, Eagle-Picher Industries, Inc., Joplin, Missouri, September 1971; prepared for NASA/GSFC, Contract Number NAS5-21159.

2) Third Quarterly Report, Ibid, June 1971.

3) First Quarterly Report, Ibid, May 1970.

4) Skoog, Douglas A., West, Donald M., Fundamentals of Analytical Chemistry, Holt, Rinehart and Winston, 1963.

APPENDIX A

EXPERIMENT NUMBER 1

	VARIABLE	LEVEL
1.	Specific Gravity of Nitrate	1.70
2.	Free Acid (Controlled By Addition of HNO_3)	1.0 gms/liter
3.	Temperature of Nitrate (In I/P Tank)	132 to 137°F
4.	Time of Impregnation	1 Hour
5.	Vacuum	15 Inches
6.	Wash Time	30 Minutes
7.	Wash (Number of Cycles)	1 Cycle
8.	Wash Water Temperature	59°F
9.	pH of Wash Water (Measured pH Paper)	4.5 to 5.3
10.	Type of Caustic	NaOH
11.	Specific Gravity of Caustic	1.30
12.	Temperature of Caustic (In I/P Tank)	64 to 74°F
13.	Amount of NH_3 in Caustic	0.021 to 0.037 N
14.	Amount of CO_3 in Caustic	0.2 N
15.	Amount of OH in Caustic	9.2 N
16.	Polarization Current	0.4 amps/in^2
17.	Polarization Time	1 Hour
18.	Voltage of Plaque to Reference	See Appendix
19.	Amount of Cycles with Same Caustic	1 Cycle
20.	Number of Total Cycles	4 Cycles
21.	Type of Plaque	3 Types

EXPERIMENT NUMBER 5A

	VARIABLES	LEVEL
1.	As shown for previous experiments	1.70
2.	" " " " "	0.80 to 1.0 gm/liter
3.	" " " " "	178 to 186°F
4.	" " " " "	1 Hour
5.	" " " " "	0 Inches
6.	" " " " "	10 Minutes
7.	" " " " "	3 Cycles
8.	" " " " "	50 to 52°F
9.	" " " " "	4.5
10.	" " " " "	NaOH
11.	" " " " "	1.30
12.	" " " " "	150 to 160°F
13.	" " " " "	0.007 to 0.018 N
14.	" " " " "	0.28 to 0.44 N
15.	" " " " "	9.4 to 9.8 N
16.	" " " " "	0.4 amp/in^2
17.	" " " " "	1 Hour
18.	" " " " "	See Appendix
19.	" " " " "	4 Cycles
20.	" " " " "	4 Cycles
21.	" " " " "	3 Types

EXPERIMENT NUMBER 6A

	VARIABLE	LEVEL
1.	As shown for previous experiments	1.80
2.	" " " " "	3.8 gms/liter
3.	" " " " "	182 to 190°F
4.	" " " " "	1 Hour
5.	" " " " "	0 Inch
6.	" " " " "	10 Minutes
7.	" " " " "	3 Cycles
8.	" " " " "	48 to 50°F
9.	" " " " "	4.5
10.	" " " " "	NaOH
11.	" " " " "	1.30
12.	" " " " "	150 to 160°F
13.	" " " " "	0.009 to 0.024 N
14.	" " " " "	0.16 to 0.24 N
15.	" " " " "	9.6 to 9.9 N
16.	" " " " "	0.4 amps/in^2
17.	" " " " "	1 Hour
18.	" " " " "	See Appendix
19.	" " " " "	4 Cycles
20.	" " " " "	4 Cycles
21.	" " " " "	3 Types

EXPERIMENT NUMBER 9 B

	VARIABLE	LEVEL
1.	As shown for previous experiments	1.80
2.	" " " " "	1.0 to 1.2 gms/liter
3.	" " " " "	186 to 190°F
4.	" " " " "	1 Hour
5.	" " " " "	0 Inch
6.	" " " " "	10 Minutes
7.	" " " " "	3 Cycles
8.	" " " " "	49°F
9.	" " " " "	4.5
10.	" " " " "	NaOH
11.	" " " " "	1.30
12.	" " " " "	138 to 156°F
13.	" " " " "	0.012 to 0.014 N
14.	" " " " "	0.32 to 0.36 N
15.	" " " " "	9.2 to 9.5 N
16.	" " " " "	0.4 amp/in^2
17.	" " " " "	15 Minutes
18.	" " " " "	See Appendix
19.	" " " " "	4 Cycles
20.	" " " " "	4 Cycles
21.	" " " " "	3 Types

APPENDIX B

EXPERIMENT NUMBER 2

	VARIABLES	LEVEL
1.	As shown for previous experiments	1.90
2.	" " " " "	0.5 to 0.6 gms/liter
3.	" " " " "	132 to 140°F
4.	" " " " "	1 Hour
5.	" " " " "	0 Inches
6.	" " " " "	10 Minutes
7.	" " " " "	3 Cycles
8.	" " " " "	50°F
9.	" " " " "	4.5
10.	" " " " "	NaOH
11.	" " " " "	1.30
12.	" " " " "	78 to 94°F
13.	" " " " "	0.007 N
14.	" " " " "	0.16 N
15.	" " " " "	8.3 N
16.	" " " " "	0.4 amps/in^2
17.	" " " " "	15 Minutes
18.	" " " " "	See Appendix
19.	" " " " "	3 Cycles
20.	" " " " "	3 Cycles
21.	" " " " "	3 Types

EXPERIMENT NUMBER 3

	VARIABLES	LEVELS
1.	As shown for previous experiments	1.80
2.	" " " " "	0.4 to 0.6 gms/liter
3.	" " " " "	130 to 138°F
4.	" " " " "	1 Hour
5.	" " " " "	0 Inches
6.	" " " " "	10 Minutes
7.	" " " " "	3 Cycles
8.	" " " " "	48°F
9.	" " " " "	4.5
10.	" " " " "	NaOH
11.	" " " " "	1.30
12.	" " " " "	92 to 103°F
13.	" " " " "	0.009 N
14.	" " " " "	0.20 to 0.92 N*
15.	" " " " "	9.0 to 9.3 N
16.	" " " " "	0.4 amps/in^2
17.	" " " " "	1 Hour
18.	" " " " "	See Appendix
19.	" " " " "	3 Cycles
20.	" " " " "	3 Cycles
21.	" " " " "	3 Types

*Suspect high carbonate indication is the result of the use of carbonate contaminated deionized water.

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